PREPARATION AND CHARACTERIZATION OF POLYPROPYLENE / PALM FIBER COMPOSITES

BY

Sarfraz Haider Abbasi

A Thesis Presented to the

DEANSHIP OF GRADUATE STUDIES

KING FAHD UNIVERSITY OF PETROLEUM & MINERALS

DHAHRAN, SAUDI ARABIA

In Partial Fulfillment of the

Requirements for the Degree of

MASTER OF SCIENCE

In

Chemical Engineering

December, 2003

King Fahd University of Petroleum & Minerals

DHAHRAN 31261, SAUDI ARABIA

DEANSHIP OF GRADUATE STUDIES

This thesis, written by Sarfraz Haider Abbasi under the direction of his thesis advisor and approved by his thesis committee, has been presented to and accepted by the Dean of Graduate Studies, in partial fulfillment of the requirements for the degree of MASTER OF SCIENCE IN CHEMICAL ENGINEERING.

Thesis Committee

B. A. Sharkh

Dr. Basel Abu Sharkh
(Thesis Advisor)

Dr. Ramazan Kahraman
(Co-Advisor)

A. Al Amer

Prof. Mohamed B. Amin
(Department Chairman)

Prof. Adnan M. Jarallah
Al-Amer (Member)

Prof. Osama Ahmed Jannadi
(Dean of Graduate Studies)

Sk. Asraf Ali

Prof. Shaikh Ashraf Ali
(Member)

Dr. Ibnelwaleed A. Hussein
(Member)

7/12/2003
Date

DEDICATION

This work is dedicated to my parents.

ACKNOWLEDGMENT

In the name of ALLAH, the Most Beneficent, the Most Merciful.

I take this opportunity with joy and fulfillment to thank the people that have contributed to the successful completion of my period of studentship at King Fahd University of Petroleum & Minerals. First I would like to show my gratitude towards ALLAH Almighty. This all was possible only because of his will.

I would like to express my thankfulness for my Thesis Advisor, Dr. Basel Abu Sharkh for his inspirational guidance, help and supervision of the thesis work. Working with him was indeed a wonderful and learning experience, which I thoroughly enjoyed. I am also very indebted to my Thesis Co-Advisor Dr. Ramazan Kahraman for his tremendous help, especially during experimental design and thesis correction. His continuous encouragement and numerous discussions helped enormously during experimentation and thesis writeup. I owe special thanks to my thesis committee members, Dr. Adnan M. Jarallah Al-Amer, Dr. Shaikh Ashraf Ali and Dr. Ibnelwaleed Hussein for their contribution and their critical review of the thesis.

I am thankful to the chairman of the Chemical Engineering Department, Dr. Mohammed B. Amin for his help and cooperation. I am also thankful to all Faculty and Staff members, specially Mr. Raisat, for their cooperation. My thanks are also due to all the staff and technicians who helped me during my thesis. Specially Mr. Alim Rizvi, Abdul Alim and Mr. Arab.

I would like to thank all my collegues and friends, specially Mr. Arshad Raza for his special antics and jolly character and Rehan Chaudry for all the sports activities and fun time together.

Finally, I offer my sincere thanks to my father, mother, sister and brothers for their love and affection and for enduring the distance from me during my studies.

TABLE OF CONTENTS

TABLE OF CONTENTS v

LIST OF TABLES vii

LIST OF FIGURES viii

ABSTRACT xiv

ARABIC ABSTRACT xv

CHAPTER ONE 1
INTRODUCTION 1
1.1 Introduction 1
1.2Background 4
1.2.1 Definitions 4
1.2.2 Constituents of Composites 6
1.2.3 Interfaces and Interphases 7
1.2.4 Distribution of Constituents 8
1.2.5 Classification 10
1.2.6 Fiber-Matrix Composites 11
1.2.7 Composition 12
1.2.8 The Matrix 12
1.2.9 The Bonding Phase 13
1.2.10 Lignocellulosic Fiber Polymer Composites 14
1.2.11 Composites with High Thermoplastic Content 16
1.2.12 Composites with Low Thermoplastic Content 18

CHAPTER TWO 20
LITERATURE SURVEY 20

CHAPTER THREE 28
EXPERIMENTAL 28
3.1 Materials 28
3.2 Fiber Preparation 30
3.3 Fiber Cleaning 33
3.4 Mixing 33
3.5 Molding 34
3.6 Composite Characterization 34
3.6.1 Mechanical Testing 34
3.6.2 SEM Study 34

3.6.3 DSC Study 35
3.6.4 FTIR Study 35

CHAPTER FOUR **36**
RESULTS AND DISCUSSION 36
4.1 Preliminary Tests 36
4.2 Effects of Fiber (Size and Content) and Use of Compatibilizers on Composite Performance 53
4.3 Morphological Characterization 94
4.4 DSC Study 102
4.5 FTIR Study 104

CHAPTER FIVE **113**
CONCLUSION 113

CHAPTER SIX **115**
RECOMMENDATIONS 115

REFERENCES **116**

APPENDICES **120**
Section A 121
Section B 125

VITA **135**

LIST OF TABLES

Table 3.1 Polypropylene (LADENE PP570P) Data Sheet 29

Table 3.2 Epolene E-43 Wax Data Sheet..... 29

Table 3.3 Epolene G-3003 Polymer Data Sheet 29

LIST OF FIGURES

Figure (1.1) The different constituents forms in composites. (Figure taken from "Composite Materials Handbook", Schwartz, 1992.).....6

Figure (1.2) Makeup of interface between fiber and matrix. (Figure taken from "Composite Materials Handbook", Schwartz, 1992.).....8

Figure (1.3) Gradient Composites. (Figure taken from "Composite Materials Handbook", Schwartz, 1992).....9

Figure (1.4) Orientation of Fibers. (Figure taken from "Composite Materials Handbook", Schwartz, 1992).....10

Figure (1.5) Classes of Composites. (Figure taken from "Composite Materials Handbook", Schwartz, 1992.....11

Figure (3.1) Length of fiber vs percentage frequency of large fiber length distribution.....31

Figure (3.2) Length of fiber vs percentage frequency of small fiber length distribution.....32

Figure (4.1) Tensile Strength of polypropylene with 30% palm fiber vs rpm with a mixing time of 8 min at 200°C. 38

Figure (4.2) Elongation at break of polypropylene with 30% palm fiber vs rpm with a mixing time of 8 min at 200°C. 39

Figure (4.3) Tensile Strength of polypropylene with 30% palm fiber vs mixing time with rpm at 50 and temperature at 200°C.....41

Figure (4.4) Elongation at break of polypropylene with 30% palm fiber vs mixing time with rpm at 50 and temperature at 200°C.....42

Figure (4.5) Tensile modulus of palm/polypropylene vs fiber content with mixing time 8 min, rpm at 50 and temperature at 200°C. 44

Figure (4.6) Tensile Strength of polypropylene vs fiber content with mixing time 8 min, rpm at 50 and temperature at 200°C.....45

Figure (4.7) Maximum Elongation at break of polypropylene vs fiber content with mixing time 8 min, rpm at 50 and temperature at 200°C. 46

Figure (4.8) Tensile Strength of palm/polypropylene composite with 30 wt% fiber vs extruder screw speed at Zone 1=195°C, Zone 2=200°C, Zone 3=205°C, and Nozzle=210°C.. 49

Figure (4.9) Tensile Elongation of palm/polypropylene composite with 30 wt% fiber vs extruder screw speed at Zone 1=195°C, Zone 2=200°C, Zone 3=205°C, and Nozzle=210°C.. 50

Figure (4.10) Tensile Modulus of palm/polypropylene composite with 30 wt% fiber vs extruder screw speed at Zone 1=195°C, Zone 2=200°C, Zone 3 =205°C, and Nozzle=210°C.. 51

Figure (4.11) Tensile Strength of palm/polypropylene composite with 30 wt% fiber vs fiber content. Extruder conditions at Zone 1=195°C, Zone 2=200°C, Zone 3=205°C, and Nozzle 210°C. Mixer conditions: mixing time 8 min, rpm at 50 and temperature at 200°C. 52

Figure (4.12) Tensile modulus vs fiber content for polypropylene reinforced with small and large palm fiber....55

Figure (4.13) Tensile strength vs fiber content for polypropylene reinforced with small and large palm fiber....56

Figure(4.14) Maximum tensile elongation vs fiber content for polypropylene reinforced with small and large palm fiber. 57

Figure (4.15) Tensile strength vs fiber content for palm fiber-polypropylene matrix composites compatibilized by 2 wt% Epolene E-43. 59

Figure (4.16) Tensile Modulus vs fiber content for palm fiber-polypropylene matrix composites compatibilized by 2 wt% Epolene E-43. 60

Figure (4.17) Tensile Elongation vs fiber content for palm fiber-polypropylene matrix composites compatibilized by 2 wt% Epolene E-43. 62

Figure (4.18) Tensile strength vs fiber content for palm fiber-polypropylene matrix composites compatibilized by 4 wt% Epolene E-43. 63

Figure (4.19) Tensile modulus vs fiber content for palm fiber-polypropylene matrix composites compatibilized by 4 wt% Epolene E-43. 64

Figure (4.20) Tensile elongation vs fiber content for palm fiber-polypropylene matrix composites compatibilized by 4 wt% Epolene E-43. 65

Figure (4.21) Tensile strength vs fiber content for palm fiber-polypropylene matrix composites compatibilized by 6 wt% Epolene E-43. 67

Figure (4.22) Tensile modulus vs fiber content for palm fiber-polypropylene matrix composites compatibilized by 6 wt% Epolene E-43. 68

Figure (4.23) Tensile elongation vs fiber content for palm fiber-polypropylene matrix composites compatibilized by 6 wt% Epolene E-43. 69

Figure (4.24) Tensile strength vs fiber content for palm fiber-polypropylene matrix composites compatibilized by 2 wt% Epolene G-3003.....70

Figure (4.25) Tensile modulus vs fiber content for palm fiber-polypropylene matrix composites compatibilized by 2 wt% Epolene G-3003.....71

Figure (4.26) Tensile elongation vs fiber content for palm fiber-polypropylene matrix composites compatibilized by 2 wt% Epolene G-3003.....72

Figure (4.27) Tensile strength vs fiber content for palm fiber-polypropylene matrix composites compatibilized by 4 wt% Epolene G-3003.....73

Figure (4.28) Tensile modulus vs fiber content for palm fiber-polypropylene matrix composites compatibilized by 4 wt% Epolene G-3003.....74

Figure (4.29) Tensile elongation vs fiber content for palm fiber-polypropylene matrix composites compatibilized by 4 wt% Epolene G-3003.....75

Figure (4.30) Tensile strength vs fiber content for palm fiber-polypropylene matrix composites compatibilized by 6 wt% Epolene G-3003.....76

Figure (4.31) Tensile modulus vs fiber content for palm fiber-polypropylene matrix composites compatibilized by 6 wt% Epolene G-3003.....77

Figure (4.32) Tensile elongation vs fiber content for palm fiber-polypropylene matrix composites compatibilized by 6 wt% Epolene G-3003.....78

Figure (4.33) Tensile modulus vs fiber content for polypropylene reinforced with large palm fibers without any treatment and with utilizing compatibilizer Epolene E-43 or G-3003 at various amounts.....80

Figure (4.34) Tensile modulus vs fiber content for polypropylene reinforced with small palm fibers without any treatment and with utilizing compatibilizer Epolene E-43 or G-3003 at various amounts.....81

Figure (4.35) Tensile strength vs fiber content for polypropylene reinforced with large palm fibers without any treatment and with utilizing compatibilizer Epolene E-43 or G 3003 at various amounts.. 82

Figure (4.36) Tensile strength vs fiber content for polypropylene reinforced with small palm fibers without any treatment and with utilizing compatibilizer Epolene E-43 or G-3003 at various amounts. 83

Figure (4.37) Tensile elongation vs fiber content for polypropylene reinforced with large palm fibers without any treatment and with utilizing compatibilizer Epolene E-43 or G-3003 at various amounts. 84

Figure (4.38) Tensile elongation vs fiber content for polypropylene reinforced with small palm fibers without any treatment and with utilizing compatibilizer Epolene E-43 or G-3003 at various amounts 85

Figure (4.39) Tensile strength vs compatibilizer content for polypropylene reinforced with palm fibers without any treatment and with utilizing compatibilizer Epolene E-43 at various amounts. 88

Figure (4.40) Tensile strength vs compatibilizer content for polypropylene reinforced with palm fibers without any treatment and with utilizing compatibilizer G-3003 at various amounts 89

Figure (4.41) Tensile modulus vs compatibilizer content for polypropylene reinforced with palm fibers without any treatment and with utilizing compatibilizer Epolene E-43 at various amounts. 90

Figure (4.42) Tensile modulus vs compatibilizer content for polypropylene reinforced with palm fibers without any treatment and with utilizing compatibilizer G 3003 at various amounts. 91

Figure (4.43) Tensile elongation vs compatibilizer content for polypropylene reinforced with palm fibers without any treatment and with utilizing compatibilizer Epolene E-43 at various amounts. 92

Figure (4.44) Tensile elongation vs compatibilizer content for polypropylene reinforced with palm fibers without any treatment and with utilizing compatibilizer G 3003 at various amounts. 93

Figure (4.45) SEM micrograph of a fractured surface of a 30 wt% fiber palm-polypropylene composite with no compatibilizer. 96

Figure (4.46) SEM micrograph of a fractured surface of a 30 wt% fiber palm-polypropylene composite with no compatibilizer. 97

Figure (4.47) SEM micrograph of a fractured surface of a 30 wt% fiber palm-polypropylene composite compatibilized with 6 wt% Epolene E-43 98

Figure (4.48) SEM micrograph of a fractured surface of a 30 wt% fiber palm-polypropylene composite compatibilized with 6wt% Epolene E-43.....99

Figure (4.49) SEM micrograph of a fractured surface of a 30 wt% fiber palm-polypropylene composite compatibilized with 6 wt% Epolene G-3003.....100

Figure (4.50) SEM micrograph of a fractured surface of a 30 wt% fiber palm-polypropylene composite with Epolene 6 wt% G-3003. 101

Figure (4.51) Effect of fiber content on fractional crystalline content of palm fiber / polypropylene composite. 103

Figure (4.52a) Transmission spectra of pure Epolene EP-43. 105

Figure (4.52b) Transmission spectra of the area of interest for Pure Epolene EP-43.....106

Figure (4.53) Transmission spectra of pure polypropylene. 107

Figure (4.54) Transmission spectra of palm fiber.....108

Figure (4.55a) Transmission spectra of Epolene G-3003. 109

Figure (4.55b) Transmission spectra of the area of interest for pure Epolene G-3003.....110

Figure (4.56) Transmission spectra of palm fiber (37%) /polypropylene (57%) with Epolene E-43 as compatibilizer. 111

Figure (4.57) Transmission spectra of palm fiber (37%) / polypropylene (57%) with Epolene G-3003 as compatibilizer.....112

Figure (A1) Sketch of Tensile Specimen.....121

Figure (A2) Pure Polypropylene (Stress/Strain curve) 122

Figure (A3) PP/20 wt% fiber (Stress/Strain Curve). 122

Figure (A4) PP/30 wt% fiber (Stress/Strain Curve) 123

Figure (A5) PP/40 wt% fiber (Stress/Strain Curve). 123

Figure (A6) 6 wt% G-3003 in PP/40 wt% fiber (SS Curve).....124

Figure (A7) 6% EP-43 added in PP/40 wt% fiber (SS Curve). 124

Figure (B1) Thermogram of Pure Polypropylene.....125

Figure (B2) Thermogram of palm fiber (20%) / polypropylene (80%) composite.....126

Figure (B3) Thermogram of palm fiber (30%) / polypropylene (70%) composite.....127

Figure (B4) Thermogram of palm fiber (40%) / polypropylene (60%) composite.....128

Figure (B5) Thermogram of palm fiber (40%) / polypropylene (60%) composite with 2% Epolene E-43.....129

Figure (B6) Thermogram of palm fiber (40%) / polypropylene (60%) composite with 4% Epolene E-43.....130

Figure (B7) Thermogram of palm fiber (40%) / polypropylene (60%) composite with 6% Epolene E-43.....131

Figure (B8) Thermogram of palm fiber (40%) / polypropylene (60%) composite with 2% Epolene G-3003.....132

Figure (B9) Thermogram of palm fiber (40%) / polypropylene (60%) composite with 4% Epolene G-3003.....133

Figure (B10) Thermogram of palm fiber (40%) / polypropylene (60%) composite with 6% Epolene G-3003.....134

ABSTRACT

Name:	SARFRAZ HAIDER ABBASI
Title:	PREPARATION AND CHARACTERIZATION OF POLYPROPYLENE / PALM FIBER COMPOSITES.
Degree:	MASTER OF SCIENCE
Major Field:	CHEMICAL ENGINEERING
Date:	May 2003

Composites of palm fiber and polypropylene were compounded using a mixing device at various temperatures, mixing times, and mixing intensities. The composites were subsequently injection molded into standard tensile specimens for mechanical characterization. The composites formed were then characterized using Mechanical Testing Machine, Scanning Electron Microscopy, Differential Scanning Calorimetry and Fourier Transform Infrared Spectroscopy. It was observed that composite modulus increases with the increase of fiber content indicating the existence of adhesion between polypropylene and the much stiffer palm fiber. However, the adhesion is not satisfactory and resulted in decrease in the composite tensile strength with fiber addition.

Two compatibilizers Epolene E-43 and Epolene G-3003 were used to minimize this incompatibility between the wood fibers and the polypropylene matrix. The additives enhanced the fiber-matrix adhesion resulting in an improvement in composite performance. Also, small fibers showed better mechanical properties compared to long fibers. Morphological characterization revealed that adding the compatibilizer gave a smooth fracture surface, due to decrease in fiber pull out which points towards chemical reaction between fiber and the compatibilizers. Diffrential scanning calorimetry showed that no significant increase in crystallinity with the addition of fiber. Fourier transform infrared spectroscopy did not conclusively show the creation of new functional groups between fiber and compatibilizer, which would have been created due to chemical reaction.

خلاصة الرسالة

الاسم: سار فراز حيدر عباسي
العنوان: تحضير وخصائص البولي بروبلين المقوى بألياف النخيل.
الدرجة: ماجستير علوم
التخصص: هندسة كيميائية
التاريخ: مايو ٢٠٠٣

تم تحضير مواد مركبة مكونة من سعف النخيل والبولي بروبلين باستخدام جهازي خلط مختلفين عند عدة درجات حرارة ، وتم تغيير وقت الخلط وكثافته . المواد المركبة تم تشكيلها على شكل عينات لدراسة الخواص الميكانيكية .المركب من ثم تمت دراستة باستخدام جهاز اختبار ميكانيكي ومجهر الكتروني ومطيافية تحت الحمراء ولقد لوحظ أن معامل القوة للمركب يزيد بازدياد محتوى الألياف مما يعني وجود الألتصاق بين البولي بروبلين ونسيج سعف النخيل ولكن قوة الالتصاق ليست قوية بشكل كاف مما نتج عن نقصان في قوة النسيج الميكانيكية مع زيادة محتوى الألياف.

تم استخدام مادتين موفقتين لتقليل عدم التوافق ما بين النسيج الخشبي والبولي بروبلين. المواد المضافة زادت من قوة الالتصاق وحسنت أداء النسيج المركب. كما أن الألياف القصيرة أظهرت خصائص ميكانيكية أفضل من الألياف الاطول. الدراسة بالمجهر الالكتروني أوضحت أن اضافة المادتين الموفقتين أعطى سطح كثير النعومة .كماأظهرت الدراسة أنه لايوجد أي تاثير على التبلور مع زيادة محتوى النسيج. بعض الطرق التي استخدمت في هدة الدراسة لم تظهر التفاعل الكيميائي في تكوين مجموعات فعالة جديدة في النسيج المركب مع المادتين المتلائمتين الدين نتجوا عن تفاعل كيميائي.

CHAPTER ONE

INTRODUCTION

1.1 Introduction

The objective of composite development is to produce a product with performance characteristics that combine the positive attributes of each constituent component. Like other lignocellulosic material, wood is strong, light weight, abundant, non hazardous, and relatively inexpensive. Any lignocellulosic can be chemically modified to enhance properties such as dimensional stability and resistance to biodegradation. This provides incentive for producing a variety of value added products from different raw materials combined to provide improvements in cost or performance, or both [Gilbert, 1994].

While there is a broad range of lignocellulosic composites, and many applications for them, they can be grouped into three general categories. The first category of lignocellulosics is that of conventional composites. Conventional composites are already in the marketplace with a high degree of customer acceptance. The lignocellulosic serves as the main ingredient, and a small percentage (generally less than 10%) of a heat-curing adhesive holds the composite together. Particleboards and fiberboards are common examples of this type of lignocellulosic composite.

The second classification is that of inorganic-bonded composites. In these composites, inorganic materials like gypsum or Portland cement hold the composite together. The lignocellulosic might be the main ingredient or serve as an aggregate. Whether used as the main ingredient of the composite or used as reinforcing filler, much of the raw material factors are the same.

The third composite classification is lignocellulosic/thermoplastic composites. In this class, the lignocellulosic can serve as reinforcing filler in a thermoplastic matrix, or conversely, the thermoplastic may serve as a binder to the lignocellulosic [Rowell et al., 1996].

Recent research is directed towards using cheap and scrap materials as reinforcing materials in thermoplastics. Rozman et al., [2000] have used coconut fibers in the formation of thermoplastic composite. Scrap shipping containers are used as source for wood filled plastic composites [Stark, 2000]. Jute has also been reported in usage for the manufacture of thermoplastic composite [Harikumar et al., 1999]. Oil palm empty fruit bunch has also been used as a reinforcing material for polypropylene composite [Rozman et al., 2001a]. Xiaoyu et al., (2001) prepared kudzu fiber reinforced polyproylene composite. They found that the melting temperature of the kudzu-PP composite was independent of the MAPP content for concentrations between 5 and 35wt%. No significant change in degree of crystallinity was observed with increasing concentration of MAPP. They also used Halpin Tsai equation to predict the tensile modulus of PP-

kudzu composite. They found that predicted values show good agreement with the experimental values up to 20% kudzu fiber loading.

In view of the above, it is the overall objective of this proposed research to investigate the feasibility in terms of mechanical properties for utilizing wood fibers obtained from waste palm tree branches in reinforcing polypropylene (a thermoplastic produced by SABIC) with a suitable compatibilizer. Parameters that influenced the properties of palm fiber thermoplastic composites were investigated. The main drive of the investigation was to develop techniques for utilizing the large amount of palm tree waste. Recent research in the field of cellulose fiber thermoplastic composites was applied to the palm fiber system. The specific objectives of the investigation were :

- Study the effects of processing conditions (temperature, mixing, residence time, fiber loading) on the dispersion and length distribution of fibers in composite.
- Study the effect of these parameters on the mechanical properties of the composite.
- Study the influence of selected group of compatibilizers and lubricant on morphological and mechanical properties of the composite.
- Investigate the relationship between properties and microstructure of the composite probed by SEM, DSC and FTIR spectroscopy.

1.2Background

The idea of combining chemical or structural elements can be productive on many different levels of matter, but for engineering purposes we must limit the concept so that we can apply it to today's problems. How can we classify the hundreds and thousands of composites? After outlining the general structural characteristics of composites, we show how this outline can be the basis of a fairly simple scheme for descriptive classification and prediction of behavior [Schwartz, 1992].

1.2.1 Definitions

There is no universally accepted definition of composite materials. Definitions in the literature differ widely. The problem is the level of definition. In the dictionary and in everyday usage the term composite refers to something made up of various parts or elements.

Elemental or basic level: At this level, that of single molecules and crystal cells, all materials composed of two or more different atoms would be regarded as composites. They would include compounds, alloys, polymers, and ceramics. Only the pure elements would be excluded.

Microstructural level: At this level of crystals, phases, and compounds, a composite would be defined as a material composed of two or more different crystals, molecular structures, or phases. By this definition many materials traditionally considered to be

monolithic or homogeneous would be classified as composites. Of all the metallic materials only single-phase alloys, such as some brasses and bronzes, would be monolithic by this definition. Steel, a multiphase alloy of carbon and iron, would be a composite.

Macrostructural level: At this level, with which we shall be principally concerned, we deal with gross structural forms of constituents, e.g., matrices, particles, and fibers, and think of a composite as a materials system composed of different macro constituents.

The definition at the macro structural level encompasses many but not all of the materials now commonly considered composites. To be more inclusive we must go beyond the forms of the constituents and include two other characteristics: (1) the individual constituents making up a composite are almost always different chemically, and (2) they are essentially insoluble in each other. A working definition of composite materials which takes into account both the structural form and composition of the material constituents follows:

A composite material is a material brought about by combining materials differing in composition or form on a macro scale for the purpose of obtaining specific characteristics and properties. The constituents retain their identity such that they can be physically identified and they exhibit an interface between one another [Schwartz, 1992].

1.2.2 Constituents of Composites

In principle, composites can be constructed of any combination of two or more materials, whether metallic, organic, or inorganic. Although the possible material combinations in composites are virtually unlimited, the constituent forms are more restricted. Major constituent forms used in composite materials are fibers, particles, laminae or layers, flakes, fillers, and matrices Figure (1.1). The matrix is the body constituent, serving to enclose the composite and give it its bulk form. The fibers, particles, laminae, flakes, and fillers are the structural constituents; they determine the internal structure of the composite. Generally, but not always, they are the additive phase.

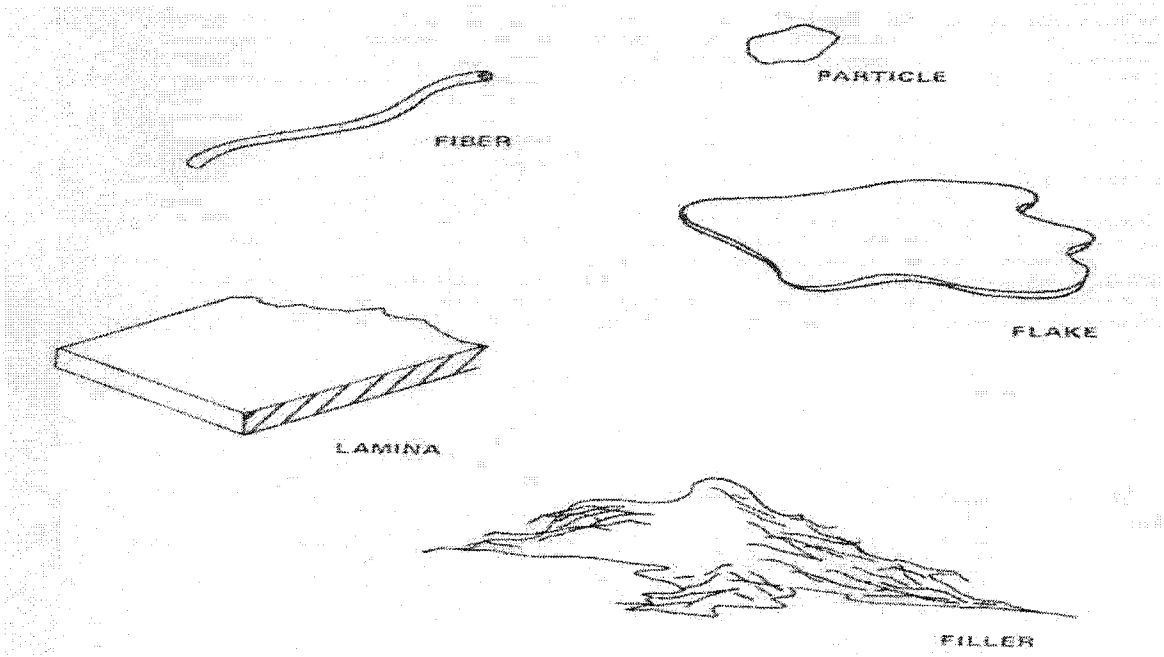


Figure (1.1) The different constituents forms in composites. (Figure taken from "Composite Materials Handbook", Schwartz, 1992.)

Perhaps the most typical composite is one composed of a structural constituent embedded in a matrix, but many composites have no matrix and are composed of one (or more)

constituent form(s) consisting of two or more different materials. Sandwiches and laminates, for example, are composed entirely of layers, which, taken together, give the composite its form. Many fabrics have no body matrix but consist entirely of fibers of several compositions, with or without a bonding phase [Schwartz, 1992].

1.2.3 Interfaces and Interphases

Because the different constituents are intermixed or combined, there is always a contiguous region. It may simply be an interface, i.e., the surface forming the common boundary of the constituents. An interface is in some ways analogous to the grain boundaries in monolithic materials. The boundary between the matrix and the reinforcement, the interface, is controlled to obtain the desired properties from a given pair of materials. In the case of ceramic composites, the interface is intentionally somewhat weak, minimizing coupling of reinforcement to matrix. When the composite is deformed, cracks propagate through the matrix until they meet a whisker. Because the interface between phases is weak, the separating matrix causes the whiskers to pull out from either side of the advancing crack front, rather than allowing the crack to continue through the whisker. The friction resulting from this pullout dissipates a large amount of energy, in addition to deflecting the crack along the fiber.

These phenomena result in substantial toughening of the composite, in some cases doubling fracture toughness relative to the unreinforced ceramic. In some cases, however, the contiguous region is a distinct added phase, called an interphase. Examples are the coating on the glass fibers in reinforced plastics and the adhesive that bonds the layers of

a laminate together. When such an interphase is present, there are two interfaces, one between each surface on the interphase and its adjoining constituent Figure (1.2).

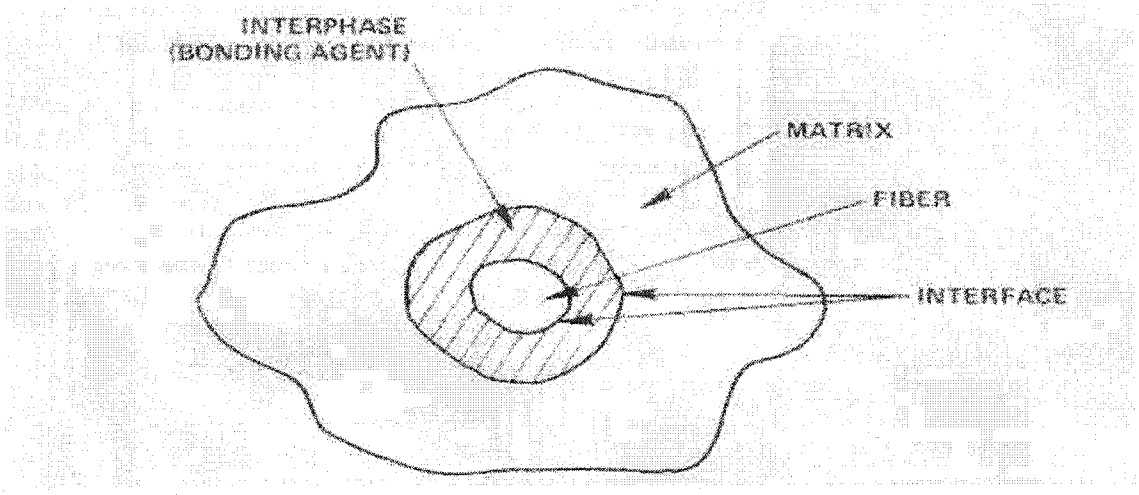


Figure (1.2) Makeup of interface between fiber and matrix. (Figure taken from "Composite Materials Handbook", Schwartz, 1992.)

The interphase of composites is the region where loads are transmitted between the reinforcement and the matrix. The extent of interaction between the reinforcement and the matrix is a design variable, and may vary from strong chemical bonding to weak frictional forces. This can be controlled by using an appropriate coating on the reinforcing fibers. Generally, a strong interfacial bond makes the composite more rigid, but brittle; a weak bond decreases stiffness, but might enhance toughness [Schwartz, 1992].

1.2.4 Distribution of Constituents

Constituents making up a composite can be distributed in two general ways. Perhaps the most common way is for the constituents to be present in a regular and repetitive pattern,

with a relatively uniform cross section both in material and structure and uniform density. Matrix-particle and some matrix-fiber composites, in which the structural constituent is evenly distributed throughout the matrix, are of this homogeneous type. The second possibility is a variable pattern of constituents that is non repetitive in either internal form or material. Materials of this type are termed graded or gradient composites. Laminated materials, which are composed of several different layers, belong in this category. Filament-wound composites can also be designed with variable fiber distribution Figure (1.3).

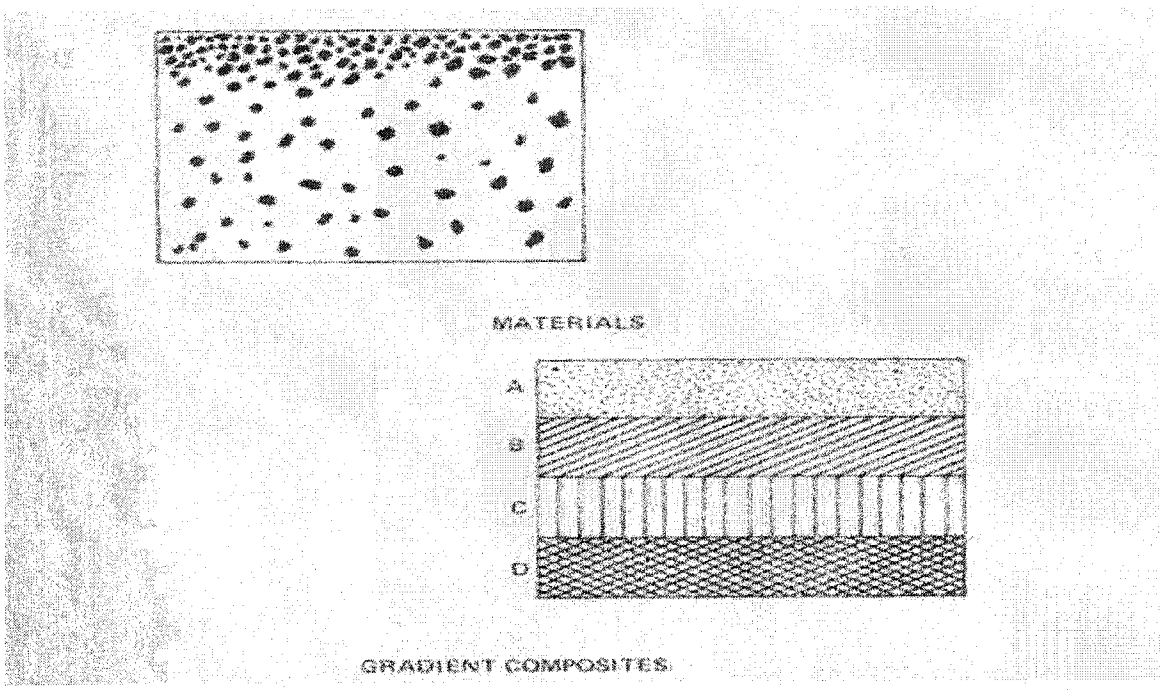


Figure (1.3) Gradient Composites. (Figure taken from "Composite Materials Handbook", Schwartz, 1992).

In both homogeneous and gradient composites the structural constituents (fibers or flakes) can be arranged in either an oriented or random fashion Figure (1.4). [Schwartz, 1992].

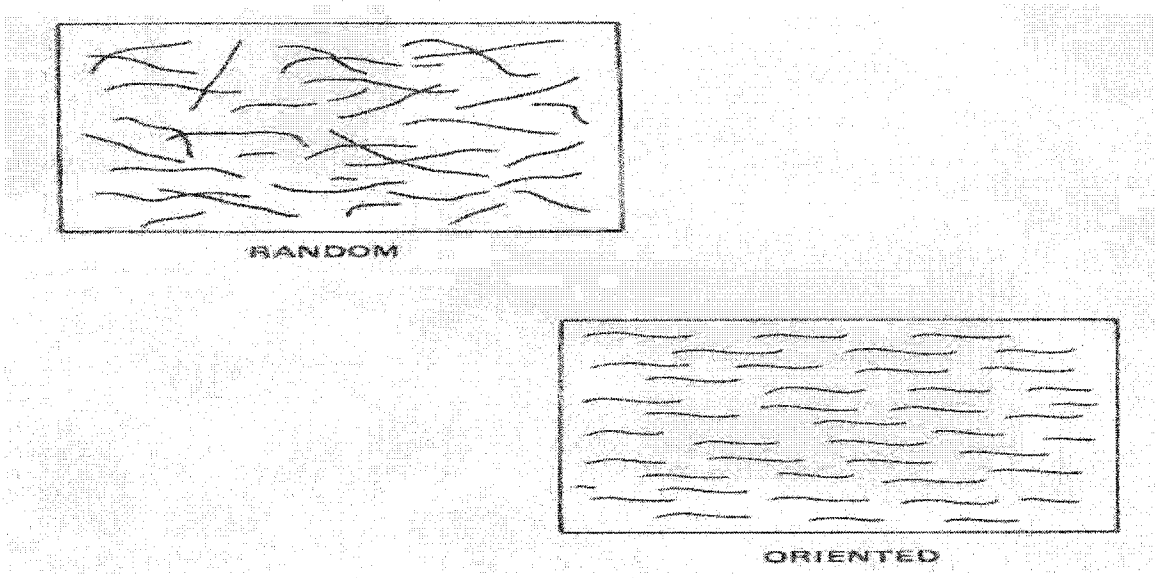


Figure (1.4) Orientation of Fibers. (Figure taken from "Composite Materials Handbook", Schwartz, 1992).

1.2.5 Classification

Several classification systems have been used, including classification (1) by basic material combinations, e.g., metal-organic or metal-inorganic; (2) by bulk-form characteristics, e.g., matrix systems or laminates; (3) by distribution of the constituents, e.g., continuous or discontinuous; and (4) by function, e.g., electrical or structural. The classification system used in this chapter is based on the form of the structural constituents. This gives five general classes of composites Figure (1.5):

1. Fiber composites, composed of fibers with or without a matrix.
2. Flake composites, composed of flat flakes with or without a matrix.
3. Particulate composites, composed of particles with or without a matrix.
4. Filled (or skeletal) composites, composed of a continuous skeletal matrix filled by a second material

5. Laminar composites, composed of layer or laminar constituents [Schwartz, 1992].

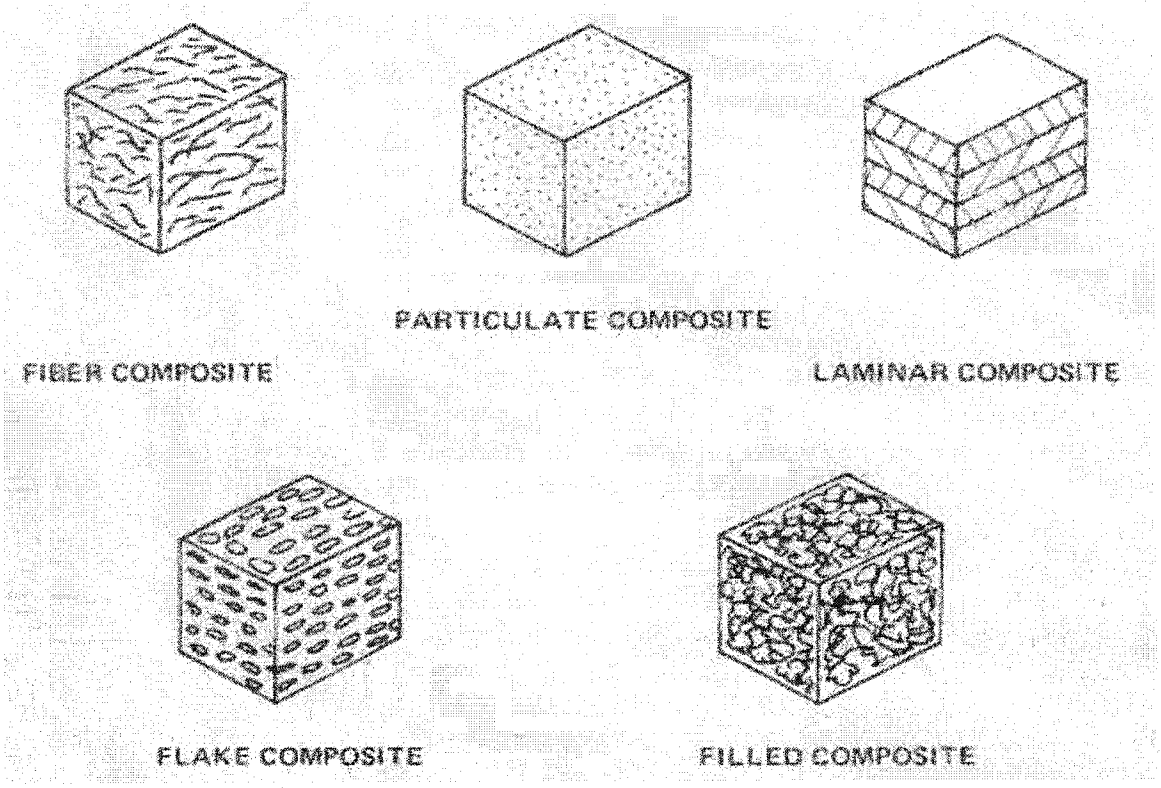


Figure (1.5) Classes of Composites. (Figure taken from "Composite Materials Handbook", Schwartz, 1992.

1.2.6 Fiber-Matrix Composites

Of all composite materials, the fiber type (specifically the inclusion of fibers in a matrix) has evoked the most interest among engineers concerned with structural applications. Initially most work was done with strong, stiff fibers of solid, circular cross section in a much weaker, more flexible matrix, i.e., glass fibers in synthetic resins. Then development work disclosed the special advantages offered by metal and ceramic fibers, hollow fibers, fibers of noncircular cross section, and stronger, stiffer, and more heat-resistant matrices [Schwartz, 1992].

1.2.7 Composition

Both organic and inorganic fibers are available for fiber composite materials. The organics, such as cellulose, polypropylene, and graphite fibers, can be characterized in general as lightweight, flexible, elastic, and heat-sensitive. Inorganic fibers, such as glass, tungsten, and ceramic, can be generally described as very high in strength, heat-resistant, rigid, and low in energy absorption as well as low in fatigue resistance. While many organic fibers satisfy both the strength and elasticity requirements for structural composites, graphite in recent years has become the most popular. The inorganics, notably glass, dominate the field. Most of the other inorganic fibers, e.g., metallic whiskers and ceramic fibers, have recently received the financial backing required for development into an accepted material [Schwartz, 1992].

1.2.8 The Matrix

The other major constituent in fiber composites, the matrix, serves two very important functions: (1) it holds the fibrous phase in place, and (2) under an applied force it deforms and distributes the stress to the high-modulus fibrous constituent. (The load is borne longitudinally by the reinforcements.) The choice of a matrix for a structural fiber composite is limited by the requirement that it have a greater elongation at break than the fiber. Thus, if the elongation at break in a fiber is 4%, the matrix must have an elongation of at least 5%, if not more. Also, the matrix must transmit the forces to the fibers and change shape as required to accomplish this, placing only tensile loading on the fibers. Furthermore, during processing the matrix must encapsulate the fibrous phase without excessive shrinkage, which can place internal strain on the fibers.

Since many reinforcements tend to be brittle, the matrix protects their surface against abrasion or environmental corrosion, both of which can initiate fracture. In order to accomplish this transfer of loads, and also reduce the chance of failure in the matrix, adhesion to fibers or other reinforcements must be coupled with sufficient matrix shear strength to sustain these loads [Schwartz, 1992].

1.2.9 The Bonding Phase

Fiber composites are able to withstand higher stresses than either of their individual constituents because the fibers and matrix interact and redistribute the stresses. The ability of these two constituents to exchange stresses depends critically on the effectiveness of the coupling or bonding between them. However, it is very common to try to maximize the coupling between fiber and reinforcement, especially for long-term property retention. It is this interfacial coupling that allows stresses to be transferred to the reinforcements dispersed through the matrix. Coupling is typically produced by wetting of the reinforcement by the matrix when it is in a molten or otherwise low-viscosity state.

Reinforcements of polymer-matrix composites are often treated with coupling agents to foster compatibility with specific resins. In fact, in many "new" fiber reinforcements, there is often nothing new about the reinforcement at all, only the coupling agent has been changed to be more compatible with a given resin system. Coupling agents are used in very small amounts, but their effects can be substantial. There are several theories on

the effect of bonding materials, variously suggesting chemical or mechanical bonding as the primary function of coupling agents

Voids (air pockets) are harmful because portions of a fiber passing through the void are not supported by surrounding resin. Under load, the fiber may buckle and transfer the stresses to the resin, which readily cracks. Weak or incomplete bonding between the fiber and the matrix is another cause of early failure. [Schwartz, 1992].

1.2.10 Lignocellulosic Fiber Polymer Composites

The use of lignocellulosics with thermoplastics is a recent innovation. Broadly defined, a thermoplastic softens when heated and hardens when cooled. Thermoplastics selected for use with lignocellulosics must melt or soften at or below the degradation point of the lignocellulosic component, normally 200°C to 220°C (392°F to 428°F). These thermoplastics include polypropylene, polystyrene, vinyls, and low- and high-density polyethylenes.

Wood flour is a readily available resource that can be used as a filler in thermoplastic composites. Wood flour is processed commercially, often from post-industrial materials such as planer shavings, chips, and sawdust. Several grades are available depending upon wood species and particle size. Wood fibers, although more difficult to process compared with wood flour, can lead to superior composite properties and act more as a reinforcement rather than a filler. A wide variety of wood fibers are available from both virgin and recycled resources.

Other materials can be added to affect processing and product performance of wood-thermoplastic composites. These additives can improve bonding between the thermoplastic and wood component (for example, coupling agents), product performance (impact modifiers, UV stabilizers, flame retardants), and processability (lubricants).

Several considerations must be kept in mind when processing wood with thermoplastics. Moisture can disrupt many thermoplastic processes, resulting in poor surface quality, voids, and unacceptable parts. Materials must either be predried or vented equipment must be used to remove moisture. The low degradation temperature of wood must also be considered. As a general rule, melt temperatures should be kept below 200°C (392°F), except for short periods. Higher temperatures can result in the release of volatiles, discoloration, odor, and embrittlement of the wood component.

There are two main strategies for processing thermoplastics in lignocellulosic composites. In the first, the lignocellulosic component serves as a reinforcing agent or filler in a continuous thermoplastic matrix. In the second, the thermoplastic serves as a binder to the majority lignocellulosic component. The presence or absence of a continuous thermoplastic matrix may also determine the processability of the composite material. In general, if the matrix is continuous, conventional thermoplastic processing equipment may be used to process composites; however, if the matrix is not continuous, other processes may be required. For the purpose of discussion, we will present these two

scenarios for composites with high and low thermoplastic content [Forest Products Laboratory, 2000].

1.2.11 Composites with High Thermoplastic Content

In composites with high thermoplastic content, the thermoplastic component is in a continuous matrix and the lignocellulosic component serves as a reinforcement or filler. In the great majority of reinforced thermoplastic composites available commercially, inorganic materials (for example, glass, clays, and minerals) are used as reinforcements or fillers. Lignocellulosic materials offer some advantages over inorganic materials; they are lighter, much less abrasive, and renewable. As a reinforcement, lingocellulosics can stiffen and strengthen the thermoplastic and can improve thermal stability compared with that of unfilled material.

Thermoplastics in pellet form have bulk density in the range of 500 to 600 kg/m^3 (31 to 37 lb/ft^3). Lignocellulosics typically have an uncompacted bulk density of 25 to 250 kg/m^3 (1.6 to 16 lb/ft^3). Wood fibers are at the low end of the lignocellulosic bulk density continuum and wood flours at the high end. Although processing of wood flour in thermoplastics is relatively easy, the low bulk density and difficulty of dispersing fibrous materials make thermoplastics more difficult to compound. More intensive mixing and the use of special feeding equipment may be necessary to handle longer fibers.

The manufacture of thermoplastic composites is usually a two-step process. The raw materials are first mixed together, and the composite blend is then formed into a product. The combination of these steps is called in-line processing, and the result is a single

processing step that converts raw materials to end products. In-line processing can be very difficult because of control demands and processing trade-offs. As a result, it is often easier and more economical to separate the processing steps.

Compounding is the feeding and dispersing of the lignocellulosic component in a molten thermoplastic to produce a homogeneous material. Various additives are added and moisture is removed during compounding. Compounding may be accomplished using either batch mixers (for example, internal and thermokinetic mixers) or continuous mixers (for example, extruders and kneaders). Batch systems allow closer control of residence time, shear, and temperature than do continuous systems. Batch systems are also more appropriate for operations consisting of short runs and frequent change of materials. On the other hand, continuous systems are less operator-dependent than are batch systems and have less batch-to-batch differences.

The compounded material can be immediately pressed or shaped into an end product while still in its molten state or palletized into small, regular pellets for future reheating and forming. The most common types of product-forming methods for wood-thermoplastic composites involve forcing molten material through a die (sheet or profile extrusion) into a cold mold (injection molding) or pressing in calenders (calendering) or between mold halves (thermoforming and compression molding).

Properties of wood-plastic composites can vary greatly depending upon such variables as type, form, and weight fractions of constituents, types of additives, and processing

history. Composites with high thermoplastic content are not without tradeoffs. Impact resistance of such composites decreases compared with that of unfilled thermoplastics, and these composites are also more sensitive to moisture than unfilled material or composites filled with inorganic material. From a practical standpoint, however, the thermoplastic component usually makes the temperature sensitivity of the composite more significant than any change in properties brought about by moisture absorption. [Forest Products Laboratory, 2000].

1.2.12 Composites with Low Thermoplastic Content

Composites with low thermoplastic content can be made in a variety of ways. In the simplest form, the thermoplastic component acts much the same way as a thermosetting resin; that is, as a binder to the lignocellulosic component. An alternative is to use the thermoplastic in the form of a textile fiber. The thermoplastic textile fiber enables a variety of lignocellulosics to be incorporated into a low-density, nonwoven, textile-like mat. The mat may be a product in itself, or it may be consolidated into a high-density product.

Experimentally, low-thermoplastic-content composites have been made that are very similar to conventional lignocellulosic composites in many performance characteristics. In their simplest form, lignocellulosic particles or fibers can be dry-blended with thermoplastic granules, flakes, or fibers and pressed into panel products.

Because the thermoplastic component remains molten when hot, different pressing strategies must be used than when thermosetting binders are used. Two options have been developed to accommodate these types of composites. In the first, the material is placed in the hot press at ambient temperature. The press then closes and consolidates the material and heat is transferred through conduction to melt the thermoplastic component, which flows around the lignocellulosic component. The press is then cooled, "freezing" the thermoplastic so that the composite can be removed from the press. Alternatively, the material can be first heated in an oven or hot press. The hot material is then transferred to a cool press where it is quickly consolidated and cooled to make a rigid panel. Some commercial nonstructural lignocellulosic-thermoplastic composites are made in this way. [Forest Products Laboratory, 2000].

CHAPTER TWO

LITERATURE SURVEY

Polymer composites, like polymers, are being extensively used in everyday life. Polymer/Graphite composites are being used in structural, aerospace and sporting goods applications [Chodak and Krupa, 2001]. Epoxy resin based composites are used as laminates for printed circuit boards, aerospace, ballistic and engineering hardware components, pollution control equipments, automotive parts, electrical components and rehabilitation products etc. However, some of its inferior characteristics viz. impact strength, resistance to weather, thermal stability, restricts the use in high performance applications [Alagar et al., 2000]. The composite of Poly (aryl ether ketone) act as a high performance material with outstanding thermal, physical and mechanical properties. Its has found application in aerospace, coating and insulating material fields [Gan et al., 2001].

Recent research has developed Polymer composite that act as solar control materials. They are required in architectural glazing to reduce the heating of building interior under solar radiation in hot weather. In particular, blocking the infrared ray properties are desirable to save energy during air conditioner usage in buildings or cars [Nagura et al., 2001]. Recently developed Polymer composite have the potential of being used as gas sensors [Okazaki et al., 1999]. Highly transparent conducting polymer composites can be

useful as antistatic coating in electronic industry where a visual inspection is necessary [Weiss and Jesus, 1998]. Conducting polymer are being blended with high strength material to overcome there poor mechanical properties [Patil et al., 2000]. These composites can also be used to shield Electromagnetic interference [Wu and Huang, 2000].

The wide spread use of plastic material has begun to create severe environmental, economic, social, and political problems. The availability of landfill space has decreased rapidly, the cost of landfilling plastics wastes has increased enormously. To alleviate these problems, plastics recycling is becoming a priority in most waste management programs [Yam et al., 1990].

The other method to tackle this problem is to use natural fibers as the reinforcements in polymers [Rozman et al., 2001a; Rozman et al., 2000; Rozman et al., 1999; Coutinho et al., 2000; Angles et al., 1999; Mwaikambo et al., 2000; Gassan et al., 2000; Harikumar et al., 1999; Xiaoyu et al., 2002]. Since natural fibers are biodegradable, the composites of natural fibers and polymers may offer a new class of materials which are environment friendly [Rozman et al., 2001a; Rozman et al., 2000; Rozman et al., 1999; Coutinho et al., 2000; Xiaoyu et al., 2002]. In addition, the low cost and high specific properties of natural fibers imply a significant property potential for the commodity synthetic polymers [Stark, 1999; Harikumar et al., 1999].

The 1990s had witnessed a burgeoning interest in the development of industrial and consumer products that combine wood and plastic. The primary impetus has come from one of these goals : 1) To reduce material costs by combining a lower cost material acting as filler or extender with a high cost material [Rozman et al., 2001a; Coutinho et al., 2000; Harikumar et al., 1999]. 2) To develop products that can utilize recycled materials and have the products themselves be recyclable; or 3) To produce a composite material that exhibits specific properties that are superior compared to either of the component materials alone (e.g. increased strength to weight ratio, improved abrasion resistance, etc) [Younguist, 1995].

The function of the plastic matrix when fibers are added is two fold. It holds the fibers in place and under applied force it deforms and distributes the stress to the high modulus fibrous constituent. Many reinforcing fibers tend to be brittle, the matrix protects their surface against abrasion or environmental corrosion, both of which can initiate fracture. In order to accomplish this transfer of loads, and also reduce the chance of failure in the matrix, adhesion to fibers or other reinforcements must be coupled with sufficient matrix shear strength to sustain this load [Quillin et al., 1994] .

Wood fibers are attractive fillers for thermoplastic composites because of low cost and large availability. They are light and provide superior physical and mechanical properties [Park and Balatinecz, 1998; Yam et al., 1990]. Xiayu et al. [2001] studied the prospect of using Kudzu (a lignocellulosic legume) as a reinforcement with polypropylene. Manrich [2000] showed in his work that synthetic paper can also be prepared from PP composite.

Faud, et al. [1995] showed that FTIR can be used as a quantitative analysis tool to quantify the filler content of polypropylene composite. Lignocellulosic material as a reinforcing component in polymer composites has received increased attention particularly for price driven and high volume applications. They offer several advantage over there inorganic counterparts, viz., lower density, greater deformability, less abrasiveness to expensive moulds and mixing equipment, and of course lower cost [Rozman et al., 2000]. Moreover, lignocellulosic based fillers are derived from renewable resources [Rozman et al., 2000; Angles et al., 1999].

The processing temperature of the cellulosic fibers in thermoplastics is limited due to the potential fiber degradation at higher temperatures. This limits their application with plastics of low melting temperatures. However, it has been reported that no deterioration of properties was observed when processing temperatures are maintained below 200°C [Stark, 1999]. Fiber composites can withstand higher stresses than either of there individual constituents because the fibers and matrix interact and redistribute the stresses. The ability of these two constituents to exchange stresses depends critically on the effectiveness of the coupling or bonding between them [Quillin et al., 1994].

It is well known that compatibility between lignocellulosic material and the polymer matrix plays a crucial role in determining the properties of a composite. Lignocellulosic materials, which have polar hydroxyl groups on the surface contributed predominately by cellulose and lignin, have difficulty in forming well bonded interface with non polar

polymer matrix such as polyethylene and polypropylene [Rozman et al., 2000; Angles et al., 1999; Manrich, 2000; Son et al., 2000].

Another drawback of lignocellulosic fillers is their high moisture absorption, result of which is swelling and decrease in mechanical properties [Angles et al., 1999]. High moisture absorbance causes swelling and a plasticizing effect resulting in dimensional instability and poor mechanical properties. These fibers are also prone to microbiological attack leading to weak fibers and reduction of life span [Mwaikambo et al., 2000]. This can be largely prevented by treating the fibers with appropriate chemical [Mwaikambo, et al., 2000] or if the hydrophilic filler is thoroughly encapsulated in an hydrophobic polymer matrix and there is good adhesion between both components [Angles et al., 1999]. However, if the adhesion level between the filler and the matrix is not good enough, diffusion pathway can pre-exist or can be created under mechanical solicitation [Angles et al., 1999].

Various chemical reagents have been employed to enhance the compatibility between the constituent materials. These include Epolene G-3002 [Rana et al., 1998], Epolene E-43 (Maelic anyhydride modified polypropylene) [Felix and Gatenholm, 1993], poly[methylene(polyphenyl isocyanate)] (PMPPIC), γ-methacryloxypropyltrimethoxysilane, poly(propylene acrylic acid) and poly(propylene-ethylene acrylic acid) [Rozman et al., 2000].

Another approach can be hybrid composites comprising of lignocellulosic fibers and glass fibers. They would provide versatility on the properties of the composite material [Rozman, et al, 2001a]. Rozman, et al. [1999] reported that properties tested for high glass fiber/low bio fibers loading are comparable with the ones with low glass fibers/high biofibers loading. So more biofibers can be incorporated in hybrid composites.

Yet another approach toward improving the properties Polymer composite with cellulose is corona treatment of the fiber. Such a treatment modifies the surface composition and, therefore, the surface properties, of the composite component. In the case of cellulose fiber, corona treatment increases the surface energy [Belgacem et al., 1994].

There are many applications of wood fiber/ plastic composite in everyday life. Wood fiber/polypropylene composite are used in cars behind the vinyl carpeting on the doors, consoles and seat backs. Many window and door manufacturers are looking at wood fiber/ plastic composites as an alternative to solid wood in clad components [Younguist, 1995].

Nafeiropoulos et al., (2001) showed that the transcrystallinity phenomenon in the flax/polypropylene system is not affected only by the different type of flax fibers but also by the different type of polypropylene. They also found that interfacial adhesion can be enhanced in flax/polypropylene without the need for sizing the interface but by simply controlling the processing conditions.

Feldman et al. [2000] found that $SiCl_4$ cold plasma treatment of PP fibers is adequate for generating very reactive Si-OH functionalities on the fiber surface and for creating rough surface topographies, and that highest tensile strength values of the composite resulting from plasma exposed PP can be related both to the formation of covalent linkages between and fibers and the matrix and to the presence of rough surface topographies .

Rozman, et al. [2001b] employed FTIR to study the evidence of chemical reaction of TDI (toluene diisocyanate) a modifier used to modify lignin which is used as compatibilizer to study Polypropylene / Empty fruit bunch fiber (EFBF) composite. They found that chemical modification of lignin with TDI imparted greater compatibility between EFBF & PP, which was reflected by improvement in both tensile and flexural strength.

Sain et al. [1993] studied polypropylene / explosion pulp composite with bimaleimide modified polyproplene and cellulose fiber. They used FTIR to quantify the adhesion between the fiber and polypropylene. They also found that percent crystallinity of polypropylene measured by DSC was found to be influenced by the presence of the modifier in the composite.

Raj et al. [1996] concluded from there work that surface treatment of kraft pulp showed an improvement in the dispersion of fibers in the polypropylene matrix. Quillin et al. [1994] showed that surface treatment of cellulose can reduce its nucleating ability in cellulose fiber/ polypropylene composite.

In view of the above, it is the overall objective of this proposed research to investigate the feasibility in terms of mechanical properties for utilizing wood fibers obtained from waste palm tree branches in reinforcing polypropylene with a suitable compatibilizer. Palm trees are abundant in the Kingdom of Saudi Arabia and polypropylene is locally produced by SABIC. If shown to be feasible, the utilization of waste palm tree branches in reinforcing thermoplastics will be an economical and useful service to the environment and society.

CHAPTER THREE

EXPERIMENTAL

3.1 Materials

Polypropylene used in the study was supplied by Saudi Basic Industries Corporation. The brand name for it is LADENE PP570P. It is a homopolymer for producing rigid injection molded articles. It is particularly suited for molding houseware items. Properties of which are given in Table 3.1.

One of the compatibilizer used in the study is Epolene E-43 supplied by Eastman Chemicals. Epolene E-43 is chemically modified polypropylene with the highest melting point of all the Epolene emulsifiable waxes. Epolene E-43 imparts excellent slip resistance and unusually good stability properties to floor finish emulsions. Due to the high melting point of Epolene E-43 wax, emulsions based on this product are most easily prepared by the direct pressure method of emulsifications. Product data sheet for Epolene E-43 is given in Table 3.2.

The other compatibilizer used is Epolene G-3003 supplied by Eastman Chemicals. Epolene G-3003 is a maleic-grafted polypropylene having optimum functionality and

molecular weight to minimize phase separation between polar reinforcement/fillers and non-polar polymers in filled composites. Its properties are given in Table 3.3.

Table 3.1 Polypropylene (LADENE PP570P) Data Sheet

Property	Unit	Value	Test Method
Flow rate (2.16 kg & 230°C)	g/10min	8	ASTM D-1238
Tensile strength @ yield	MPa	35	ASTM D-638
Tensile Elongation @ yield	%	10.5	ASTM D-638
Flexural Modulus (1% secant)	MPa	1630	ASTM D-790A
Izod Impact Resistance @ 23°C	J/m	30	ASTM D-256
Heat Distortion Temp @ 455KPa	°C	86	ASTM D-648
Hardness	Rockwell	102R	ASTM D-785

Table 3.2 Epolene E-43 Wax Data Sheet

Property	Typical Value, Units
Ring and Ball Softening Point	157°C
Density @ 25°C	0.93 g/mL
Acid Number (mg KOH/g)	47
Viscosity, Brookfield @ 190°C	400 cP
Color, Gardner	11
Molecular Weight Mn	3900

Table 3.3 Epolene G-3003 Polymer Data Sheet

Property	Typical Value, Units
Ring and Ball Softening Point	158°C
Density @ 25°C	0.912 g/mL
Acid Number (mg KOH/g)	8
Viscosity, Brookfield @ 190°C	60000 cP
Molecular Weight	
Mw	52000
Mn	27200
Yellowness Index	25

3.2 Fiber Preparation

Branches obtained from palm trees were cut into pieces about 6 in long. Thereafter they were dried in the sun for a few days so that most moisture is removed. The pieces of branches were then granulated to a small size using a Granulator (Rapid Granulator Inc, USA, Type 79-C, No 201544). The fibers were then size separated by using sieving machine. Two different size distributions of fibers were used for study. The fibers length distributions were characterized using a digital vernier caliper. The average length, standard deviation and aspect ratios for both size distributions were :

	Avg. Length (mm)	Standard Deviation (mm)	Aspect Ratio (L/D)
Large Fiber Distribution	4.77	1.75	10.86
Small Fiber Distribution	2.95	1.19	11.05

The aspect ratio is defined as the length to diameter ratio. The graphical representation of the fiber length distribution for both long and short fibers is given in Figure (3.1) and (3.2). 100 data points were used in each graph.

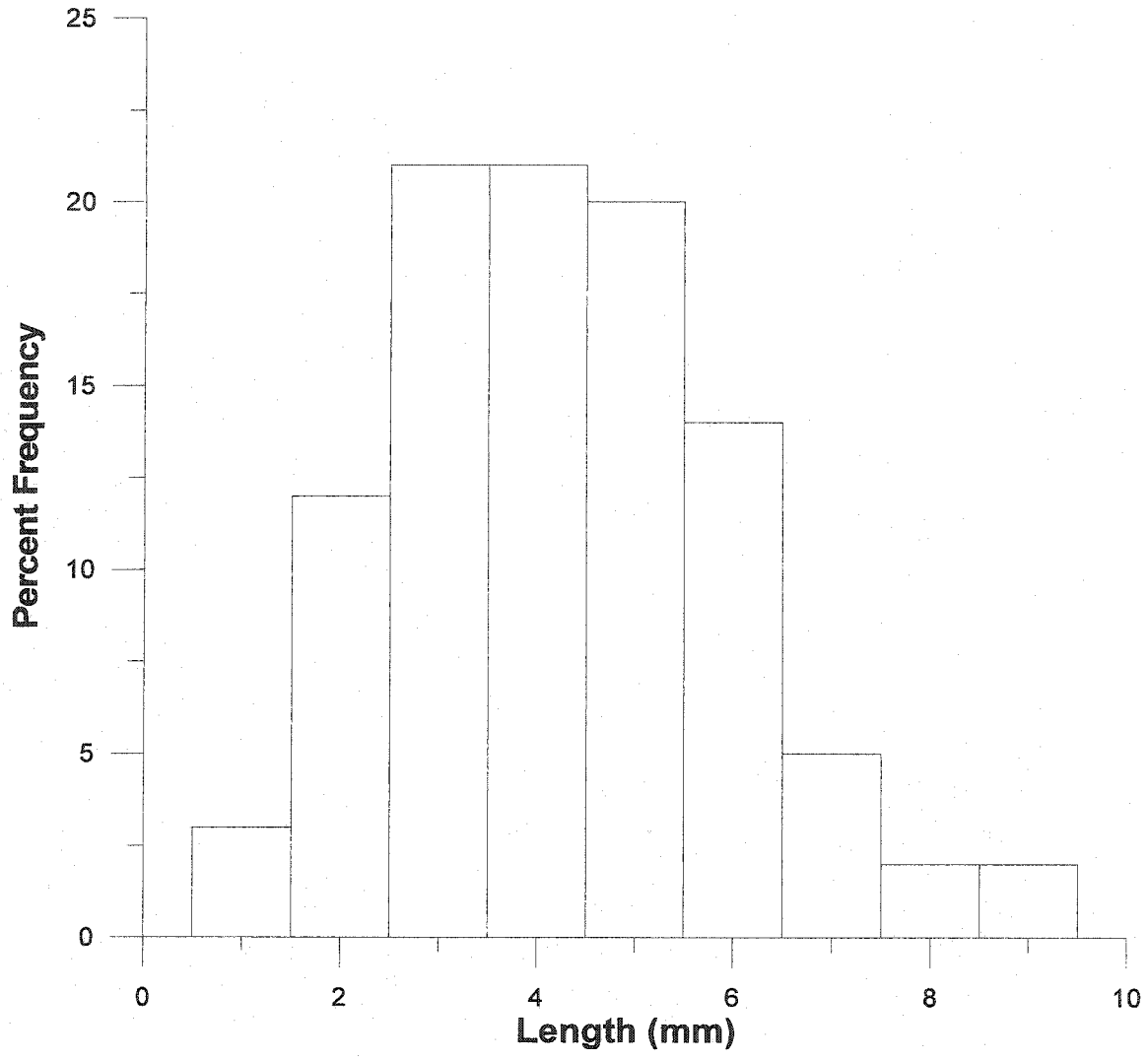


Figure (3.1) Length of fiber vs percentage frequency of large fiber length distribution.

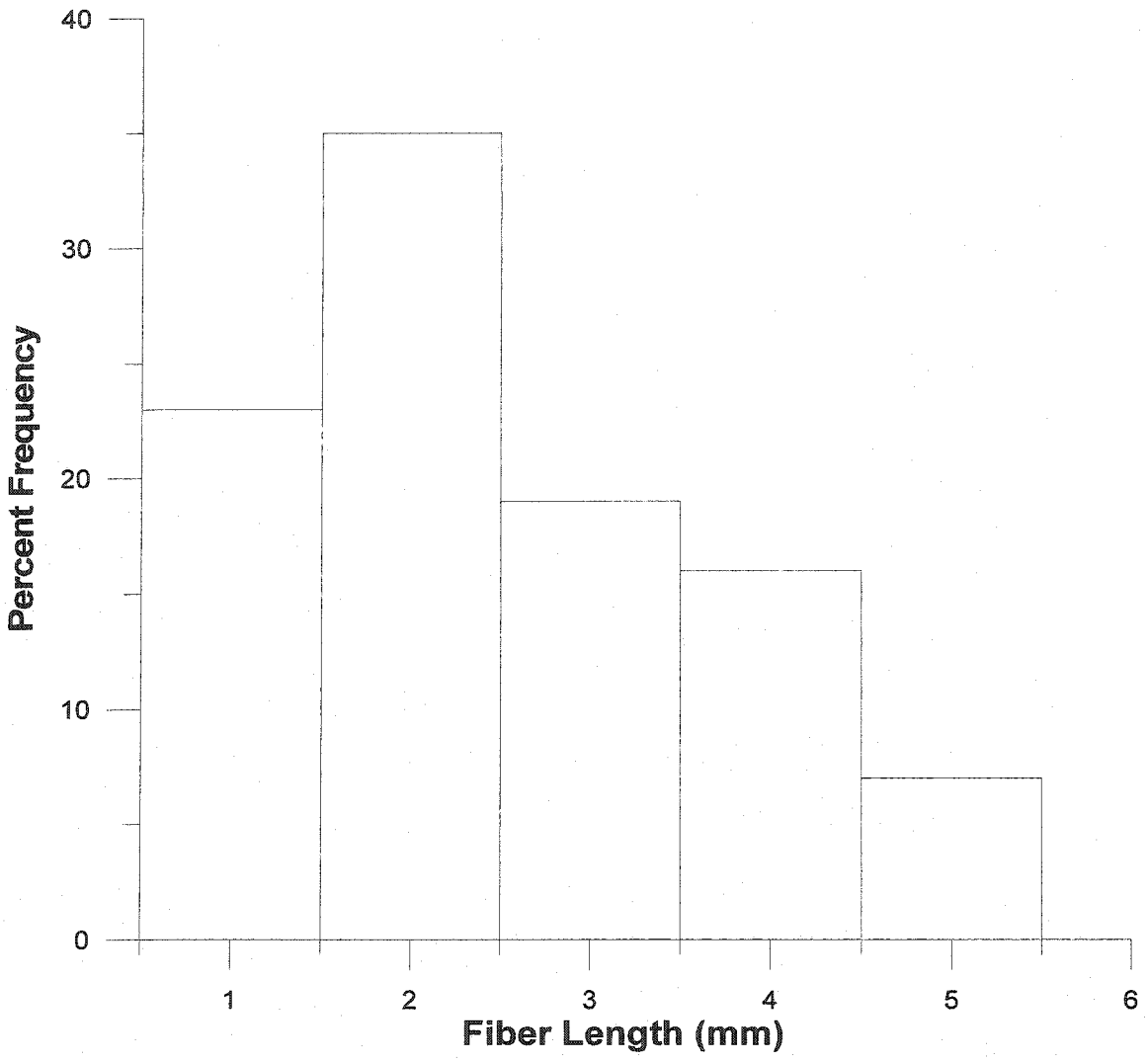


Figure (3.2) Length of fiber vs percentage frequency of small fiber length distribution.

3.3 Fiber Cleaning

The fibers obtained were then cleaned to remove the organic compounds. The chemicals used for cleaning were Ethanol and Toluene in the ratio of (1:2) (V/V). The mixture of these two chemicals was prepared in a large container. The fibers were then added to the mixture and kept in it for about two days. The fibers were then washed with water and put in an oven (Blue M Electric Company, Model OV-490A-2) at 80°C for 24 hours to remove the moisture.

3.4 Mixing

Mixing was done by a Mixing Device (Brabender Measuring Head, Model S-650, No:G126). This equipment can either be mounted with a mixer or a single screw extruder. The fibers were mixed with Polypropylene resin in the mixing device. In both cases (whether a mixer or extruder used) the fibers and the resins were first mixed by hand in a bowl and then transferred to the mixer or extruder. The conditions of mixing were varied in such a way to determine the optimum conditions that will produce samples with maximum dispersion of fibers, good mechanical properties, good color and with minimum unwanted odor. Details are given in the results section. The mixed composite thus obtained from both the mixer and the extruder had a lumpy shape. It was again granulated so that it can easily be molded. Initially experiments were done to study the reproducibility and it was found that same processing condition gave good reproducibility of results.

3.5 Molding

The granulated material was then molded with a molding machine (Engel Electronics Model ES 80/25 ST Pressure 160 bar) at 200°C. The molding machine gave us molded samples of ASTM test standard No. D-638 – 94B type II, which were then tested. Sketch of the specimen is given in Appendix [Figure (A1)].

3.6 Composite Characterization

The composite thus formed was characterized using Mechanical Testing, SEM, DSC and FTIR spectroscopy.

3.6.1 Mechanical Testing

The samples were then tested using an Instron 5560 Mechanical Testing Machine according to the ASTM test standard D-638.The tests were conducted at constant strain rate of 2 mm/min. The measured quantities were:

Maximum Strength in Mega Pascals, Modulus in Mega Pascals, Elongation in mm/mm plus data for stress/strain curve.

3.6.2 SEM Study

Scanning electron microscope (JEOL-JSM-T-300) was used to analyze the fracture surface of the composites from the tensile tests. The objective was to get information regarding the effect of the compatibilizer on fiber dispersion and adhesion quality between fibers and the matrix. JEOL-FINE COAT ION SPUTTER was used to coat a

thin layer of gold on the specimen to avoid electrostatic charging during examination. The fracture ends of the specimens were thereafter mounted on aluminum stub for study.

3.6.3 DSC Study

Differential Scanning Calorimetry was performed using METTLER TOLEDO DSC 882C STARe Thermal Analysis System. It was performed to see whether there is a change in the level of crystallinity of the composite by the addition of the compatibilizer. Percentage crystallinity was calculated using the software package of the equipment.

3.6.4 FTIR Study

FTIR study was performed using FTIR 16FPC (Perkin Elmer). The samples were prepared first by grinding the material into very small size using the grinder supplied with the FTIR equipment. Then the samples were prepared with the addition of KBr. FTIR was performed to analyze whether new functional group were formed with the addition of compatibilizer, which would attribute to the increase in mechanical properties.

CHAPTER FOUR

RESULTS AND DISCUSSION

First of all the fibers used in the composites were characterized as presented in the Experimental section. Two different fiber length distributions were used during the study. Here onwards in this thesis larger fiber length distribution with average length of 4.77 mm shall be termed as large fibers and small fiber length distribution with average length of 2.95 mm shall be called small fibers. The detail of these characterization procedures have been discussed previously. Representative Stress / Strain curves obtained during mechanical characterization are given in Appendix (Section A). As previously discussed that a mixer as well as an extruder were used for mixing. Initial tests were done on both equipment to see which equipment would suit us better in performing the experiments.

4.1 Preliminary Tests

First the mixing process was performed using the mixer mounted on the mixing device. Initial tests were performed to find out

- The effect of molding temperature on the mechanical properties of polypropylene composite.
- The effect of mixing speed (rpm) on the mechanical properties of the composite.

- The effect of mixing time on the mechanical properties of the composite.
- The effect of fiber loading on the mechanical properties of the composite.

The effect of molding temperature is quite evident even from the first samples. If the sample was molded above 200°C it was observed that the composite material burned. This can be attributed to the fact that palm fiber was being used. Below 200°C polypropylene did not melt completely.

Thereafter the effect of mixing speed on the mechanical properties was studied. From Figure (4.1) it can been seen that the highest strength of the composite was obtained at 50 rpm. This can be explained by the fact that below this rpm, adequate wetting of the fiber with the polypropylene matrix was not achieved, and at higher rpm the fiber was damaged. Same trend can be seen in Figure (4.2) for elongation at break, optimum results being obtained at 50 rpm. The processing conditions for Figures (4.1) and (4.2) were : Mixing Time: 8 minutes, Mixing Temperature: 200°C, Molding Temperature: 200°C.

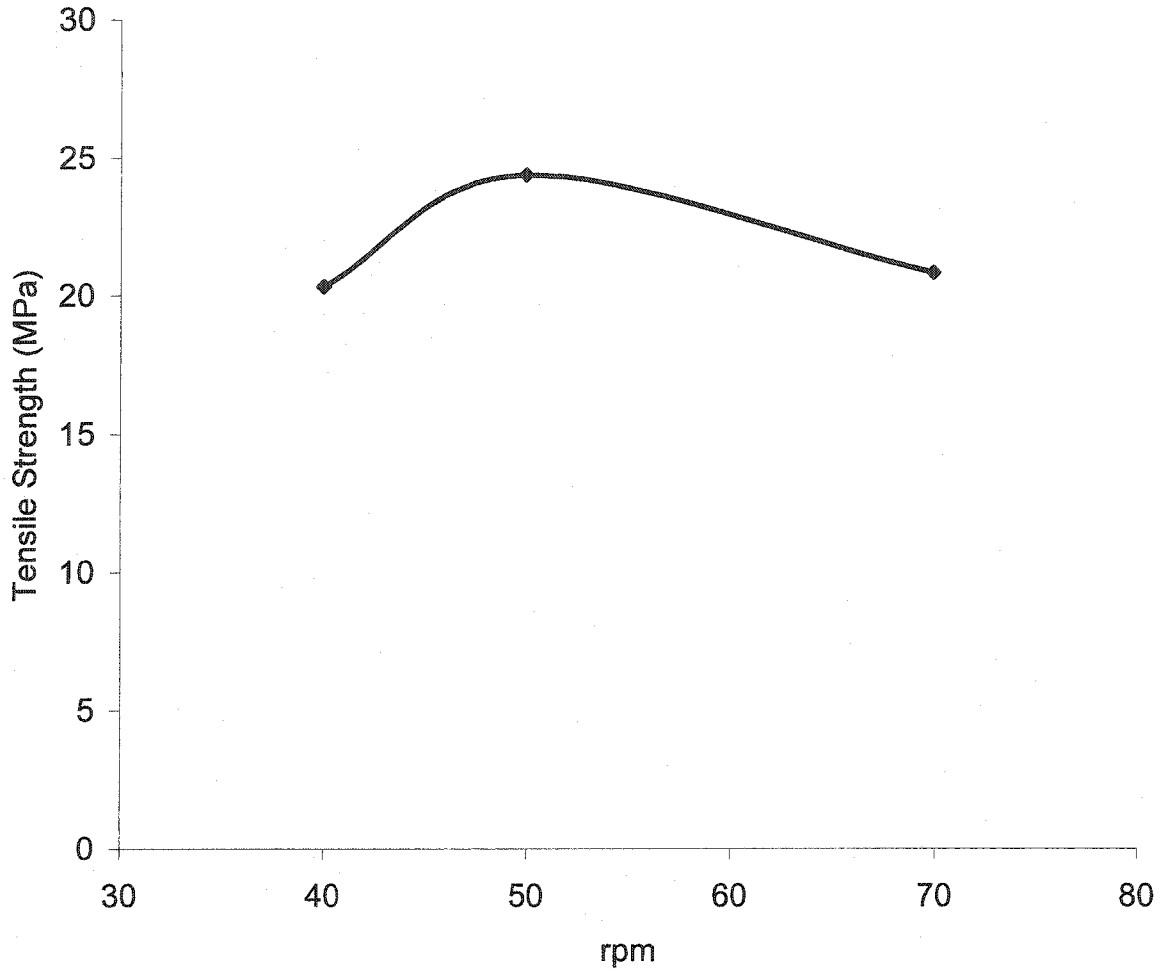


Figure (4.1) Tensile Strength of polypropylene with 30% palm fiber vs rpm with a mixing time of 8 min at 200°C.

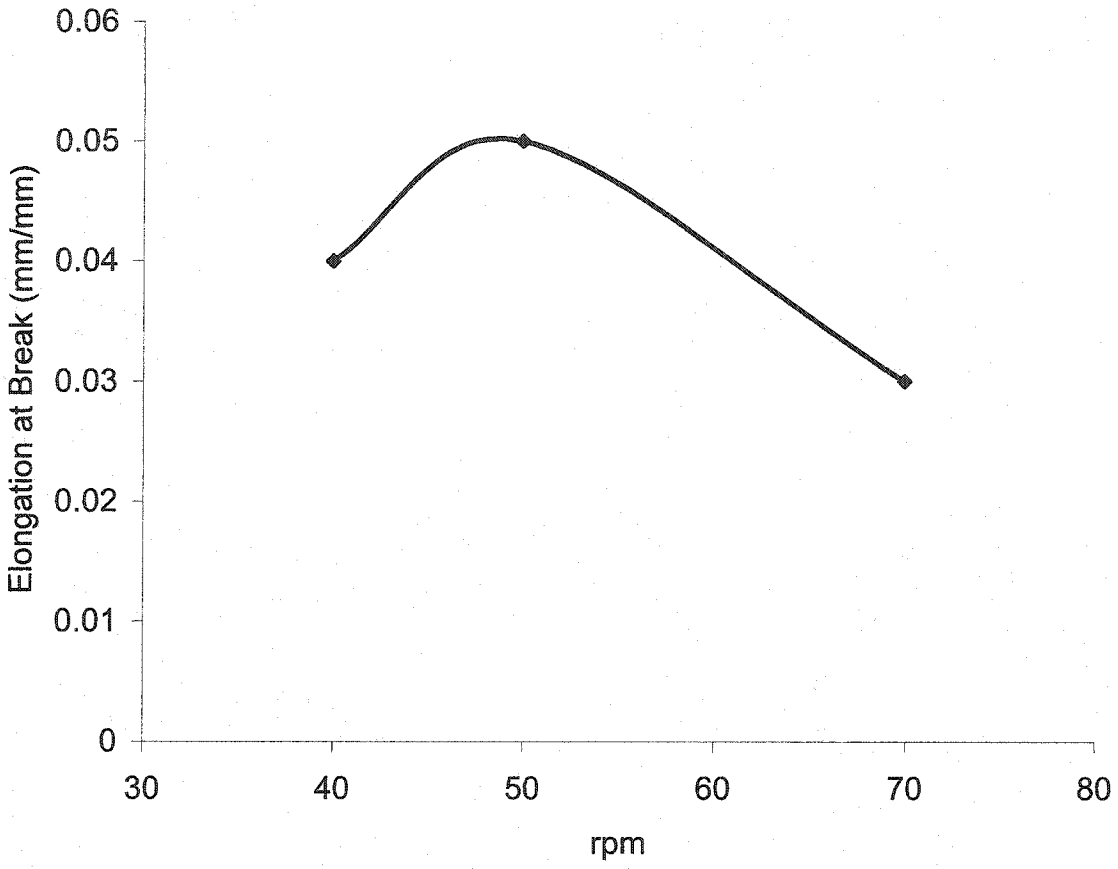


Figure (4.2) Elongation at break of polypropylene with 30% palm fiber vs rpm with a mixing time of 8 min at 200°C.

To study the effect mixing time on the mechanical properties of the polypropylene composite, the rpm was kept at 50, mixing temperature was 200°C and molding temperature was 200°C. Figures (4.3) and (4.4) show the effect of mixing time on tensile strength and elongation at break of the prepared composite. It is clear in both graphs that there is not much difference in properties for 8 and 10 min, except slight drop in maximum strength in case of 10 min. But for 15 min both the maximum strength and elongation at break decrease, which can be attributed to the fact the mixing at 200°C for too long causes the fiber to burn, the composite produced visually seemed burned as it was blackened, hence dropping the properties drastically. The conditions were 30% fiber content, 50 rpm, Mixing Temperature: 200°C, Molding Temperature: 200°C.

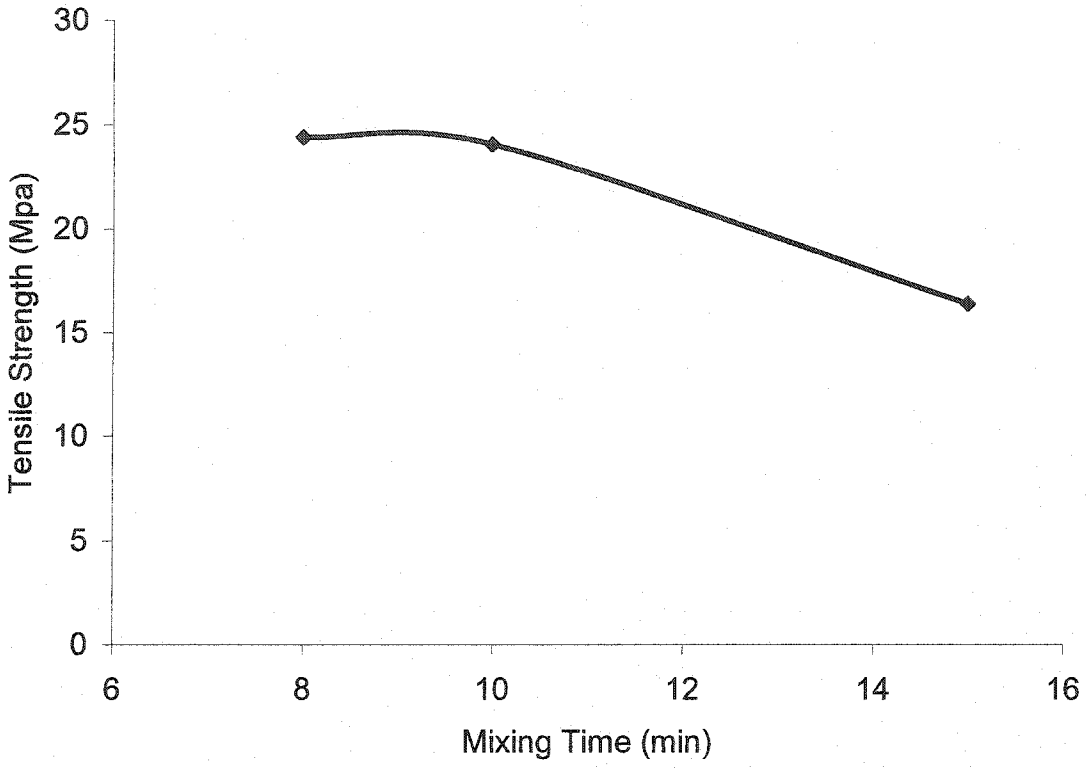


Figure (4.3) Tensile Strength of polypropylene with 30% palm fiber vs mixing time with rpm at 50 and temperature at 200°C.

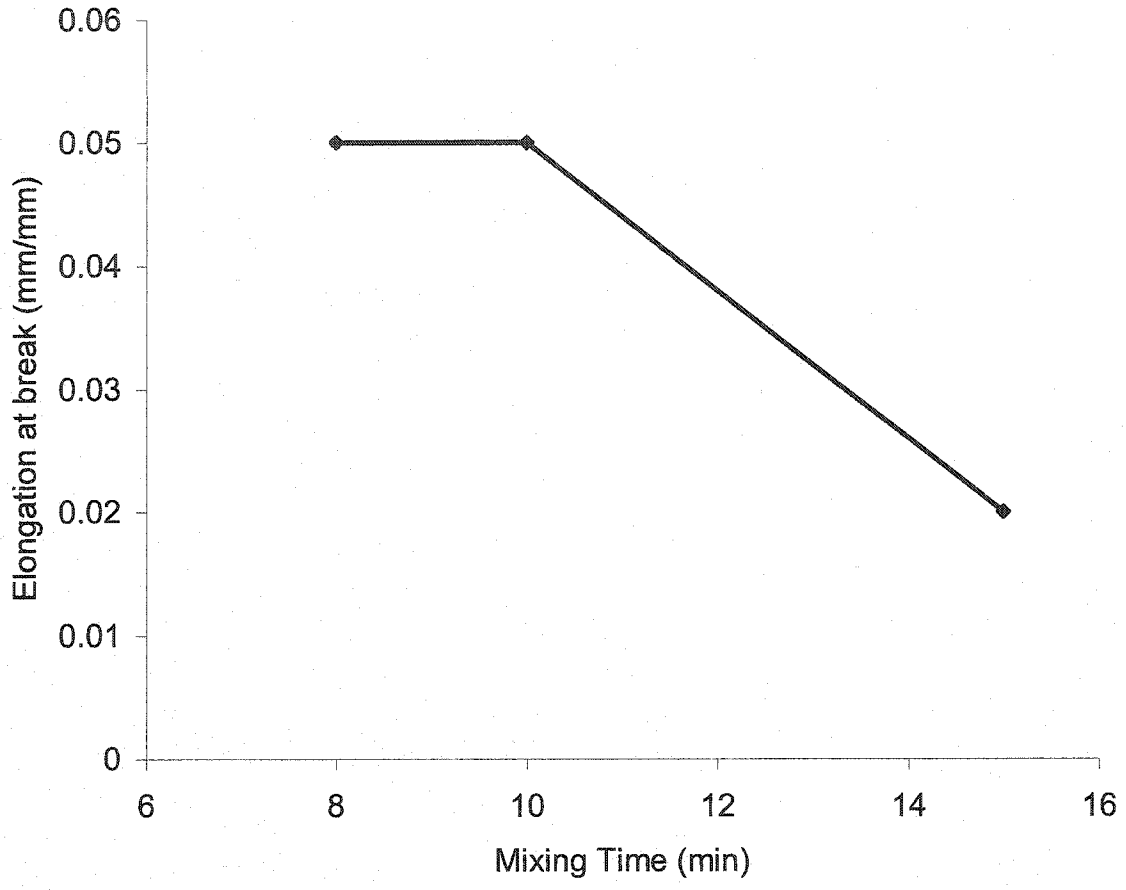


Figure (4.4) Elongation at break of polypropylene with 30% palm fiber vs mixing time with rpm at 50 and temperature at 200°C.

It can be deduced from the results presented above that the optimum rpm is 50, optimum mixing time is 8 min. After selection of the optimum conditions for mixing speed and mixing time and molding temperature, the composite were prepared under these conditions. The content of fiber was increased to see its effect on the mechanical properties. Figure (4.5) shows the effect of fiber loading on the modulus. The modulus increases with the increase in fiber loading. But maximum strength and elongation at break decreases with increase in fiber content [Figures (4.6) and (4.7) respectively]. The decrease in the strength and maximum elongation might be due to the inherent incompatibility between hydrophobic polypropylene and hydrophilic palm fiber, that is the reason why the polymer matrix cannot transmit the load to the reinforcing fibers, and/or decreased deformatibility of the matrix by rigid fiber addition [Rozman et al., 2000].

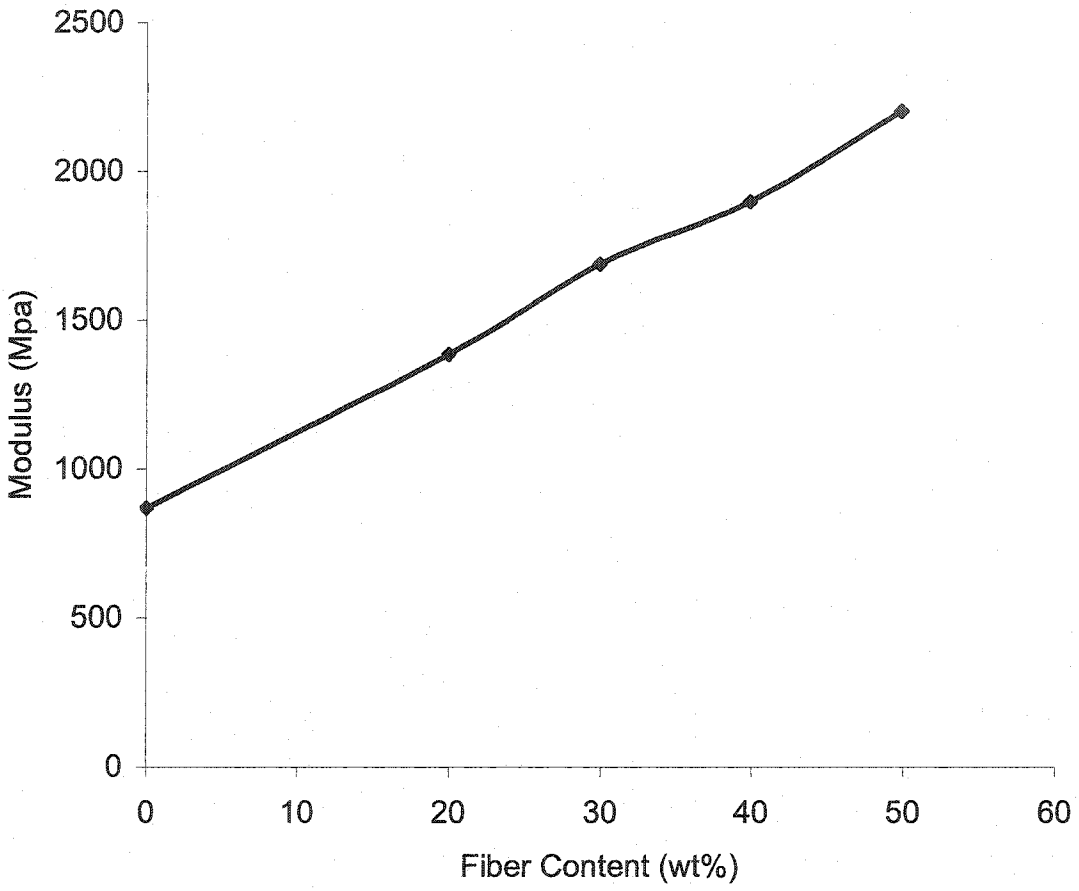


Figure (4.5) Tensile modulus of palm/polypropylene vs fiber content with mixing time 8 min, rpm at 50 and temperature at 200°C.

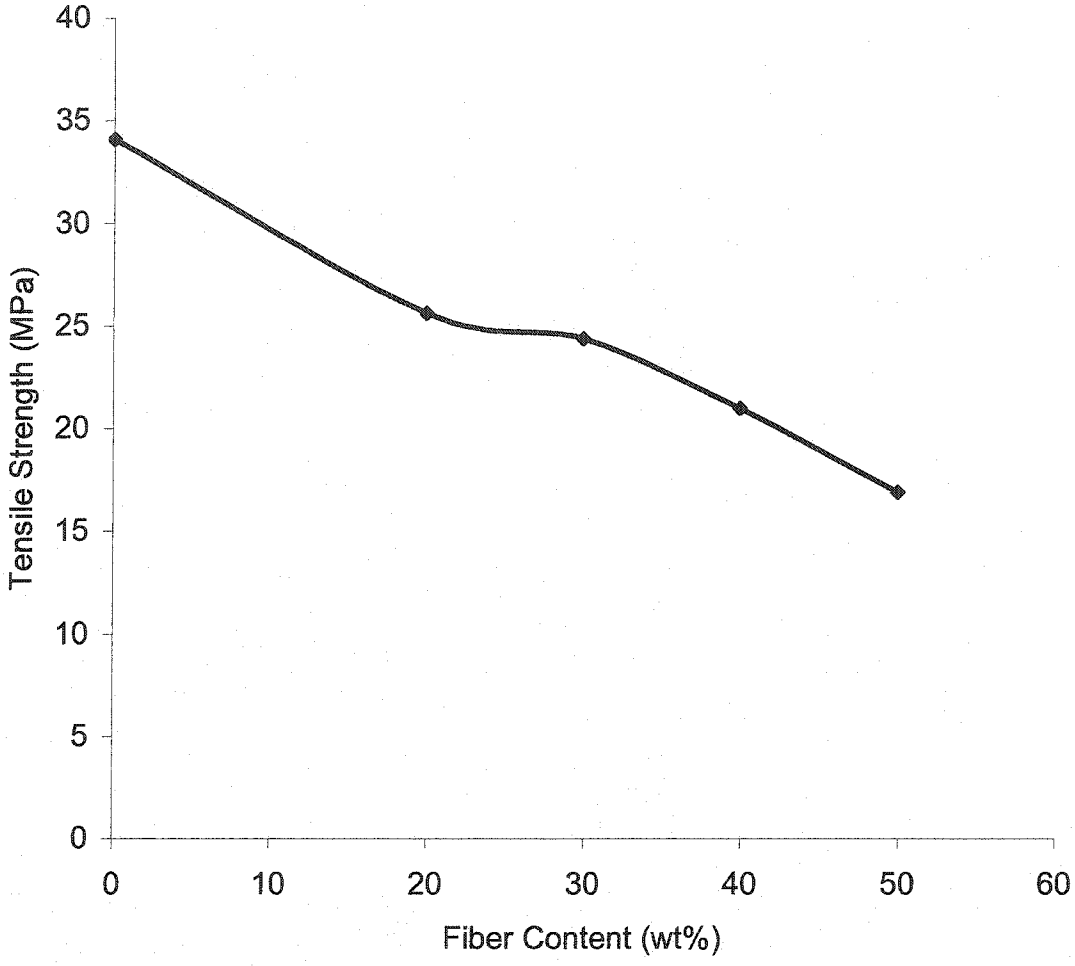


Figure (4.6) Tensile Strength of polypropylene vs fiber content with mixing time 8 min, rpm at 50 and temperature at 200°C.

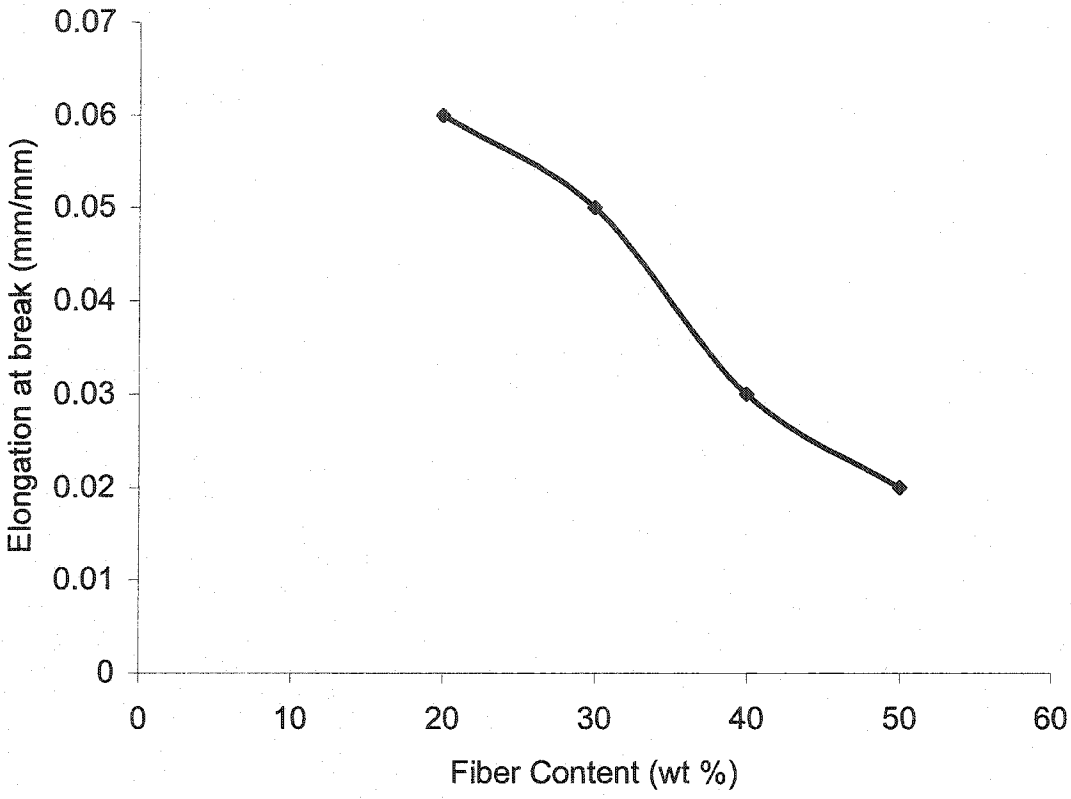


Figure (4.7) Maximum Elongation at break of polypropylene vs fiber content with mixing time 8 min, rpm at 50 and temperature at 200°C.

However from Figure (4.5) we can see as the amount of fiber is increased the modulus increases which mean that there is some adhesion. If there had been no adhesion then fibers would have acted as holes in the matrix and there would have been a decrease in the modulus. But this is not the case, so we can conclude that there is adhesion present but it is insufficient.

To study the effect of using an extruder instead of the mixer, an extruder was mounted on the mixing device, and similar tests were performed to see how the properties shall be effected by changing the equipment. The extruder had three zones and a nozzle. The temperature in the three zones and nozzle was Zone 1 =195°C, Zone 2=200°C, Zone 3=205°C, and Nozzle temperature was 210°C. If the sample were extruded above 210°C it was observed that the composite material burned. This can be attributed to the fact that palm fiber was being used. Any temperature less than 210°C in the nozzle meant a subsequent drop in temperature in previous zone. Hence the polypropylene didn't melt. There was a safety lock that used to break, if there was too much stress on the screw extruder. Hence lower temperature used to break the safety lock. Now the only parameter we can control is the rpm of the extruder. We did some test on that and the results are discussed in the graphs below.

The condition like temperature and fiber content were kept constant and only the rpm of the extruder screw was varied. The tests were conducted at temperature Zone 1=195°C, Zone 2=200°C, Zone 3=205°C, and Nozzle temperature was 210°C. The fiber content was kept constant at 30% by weight and no compatibilizer was used. Figures (4.8), (4.9)

and (4.10) shows the effect of varying the rpm on the tensile strength, tensile elongation and tensile modulus respectively. It can be seen from Figure (4.8) that there is some increase in tensile strength with increase in rpm but not significant. The rpm used here was restricted to 40 rpm because higher rpm caused the safety lock on the mixing device to break. Figure (4.9) shows that tensile elongation is somewhat increasing with the rpm. But Figure (4.10) shows a decrease in tensile modulus with increase in rpm. Hence we decided to do rest of the tests at 40 rpm, as on this rpm, two of the properties were improving, be it not a big improvement.

Now that the test for optimizing the parameter of both mixer and extruder are complete, we now compare some of their results to decide which equipment we should use. This should not only give us improved properties but also enables us to perform maximum variation of parameters.

Figure (4.11) compares the effect of fiber content on the tensile strength of the composite prepared through mixer and extruder. It is observed that at a given fiber content, the tensile strength achieved in the extruder was higher than that by the mixer. This can be attributed to the fact that the fiber spends less time at a higher temperature in an extruder (approximately 2 minutes) than in the mixer (8 minutes). Also as the residence time in the extruder was low so that average time used to mix the material was far less then when using the mixer. So it was decided that detailed tests shall be performed using the extruder.

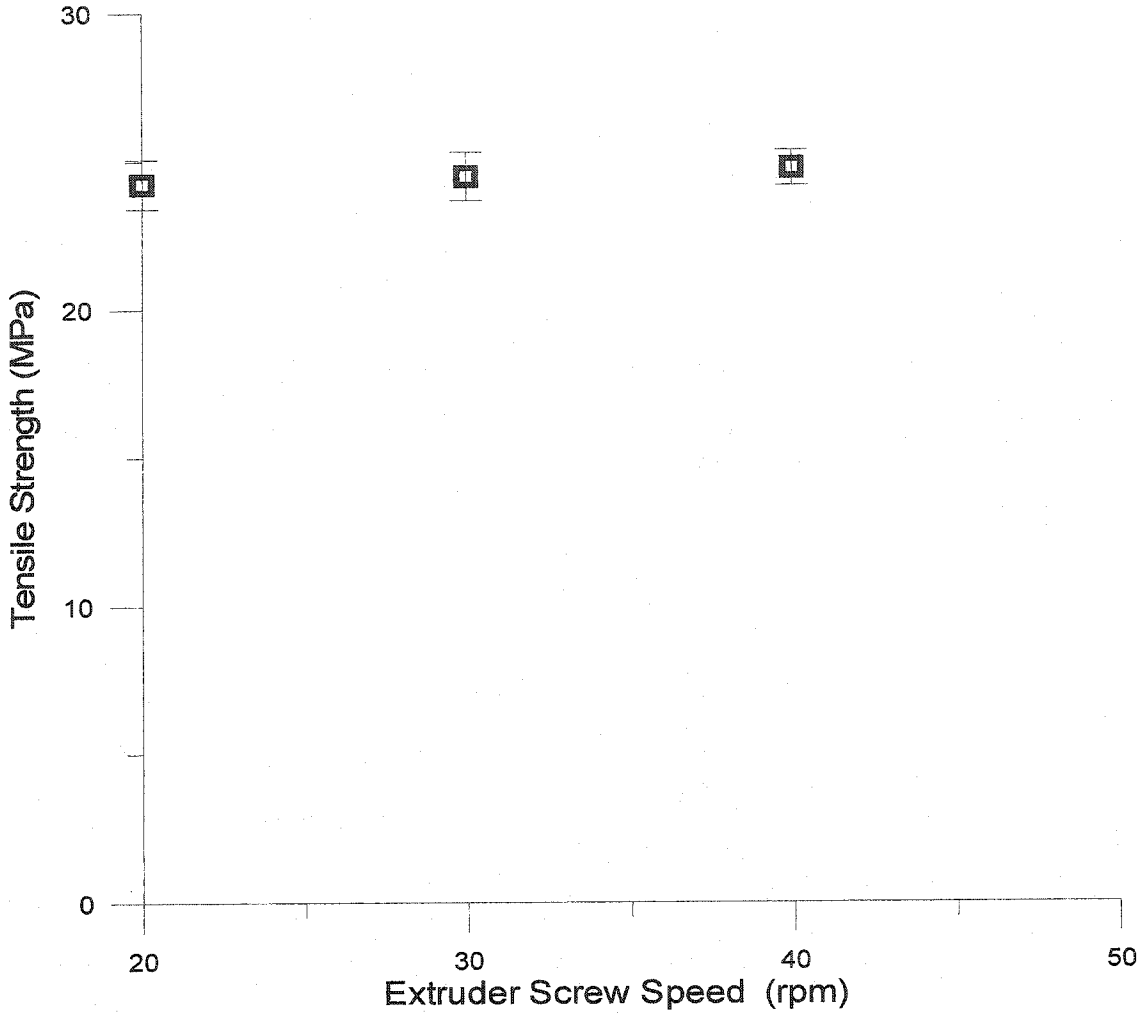


Figure (4.8) Tensile Strength of palm/polypropylene composite with 30 wt% fiber vs extruder screw speed at Zone 1=195°C, Zone 2=200°C, Zone 3=205°C, and Nozzle=210°C.

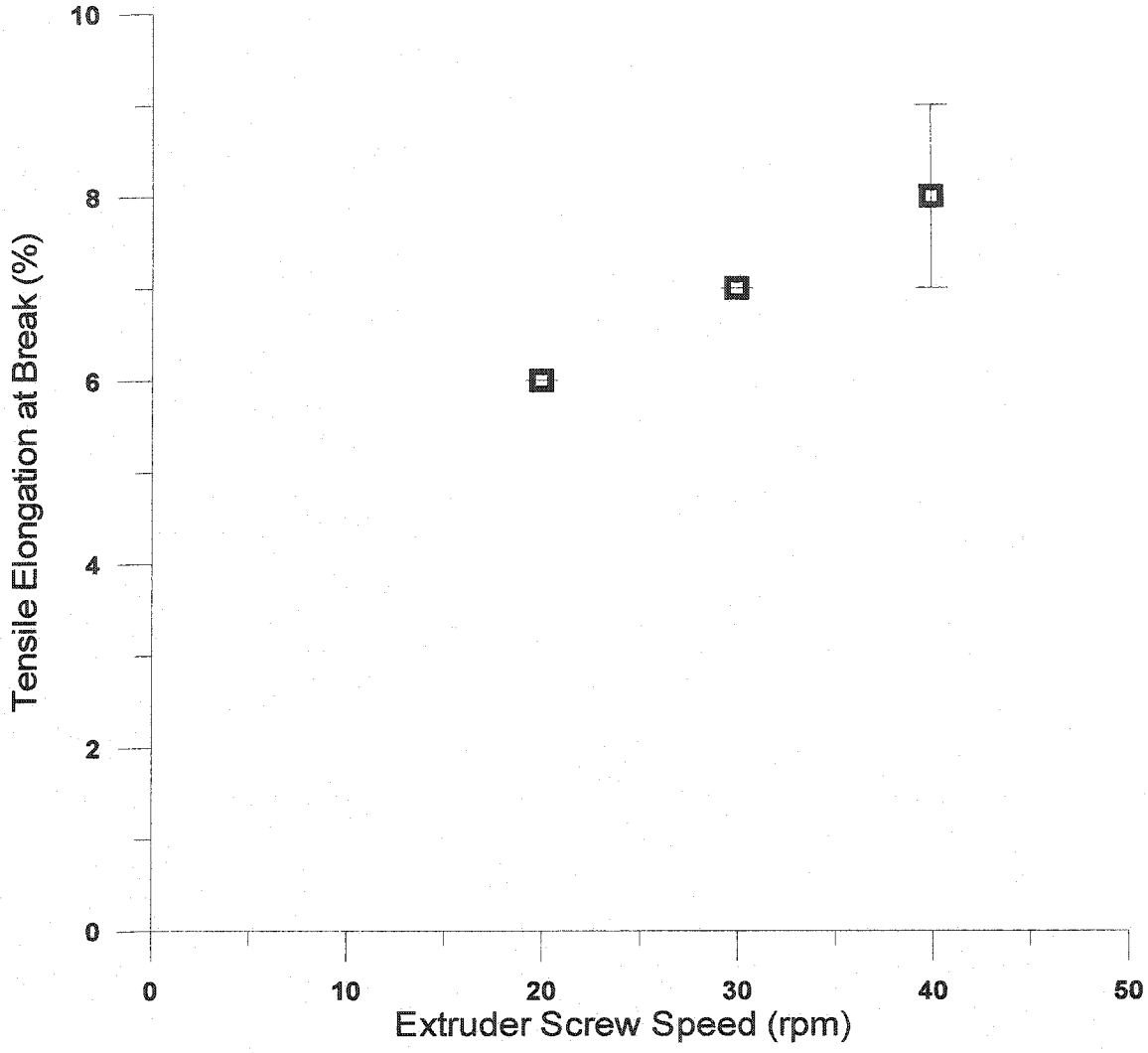


Figure (4.9) Tensile Elongation of palm/polypropylene composite with 30 wt% fiber vs extruder screw speed at Zone 1=195°C, Zone 2=200°C, Zone 3=205°C, and Nozzle=210°C.

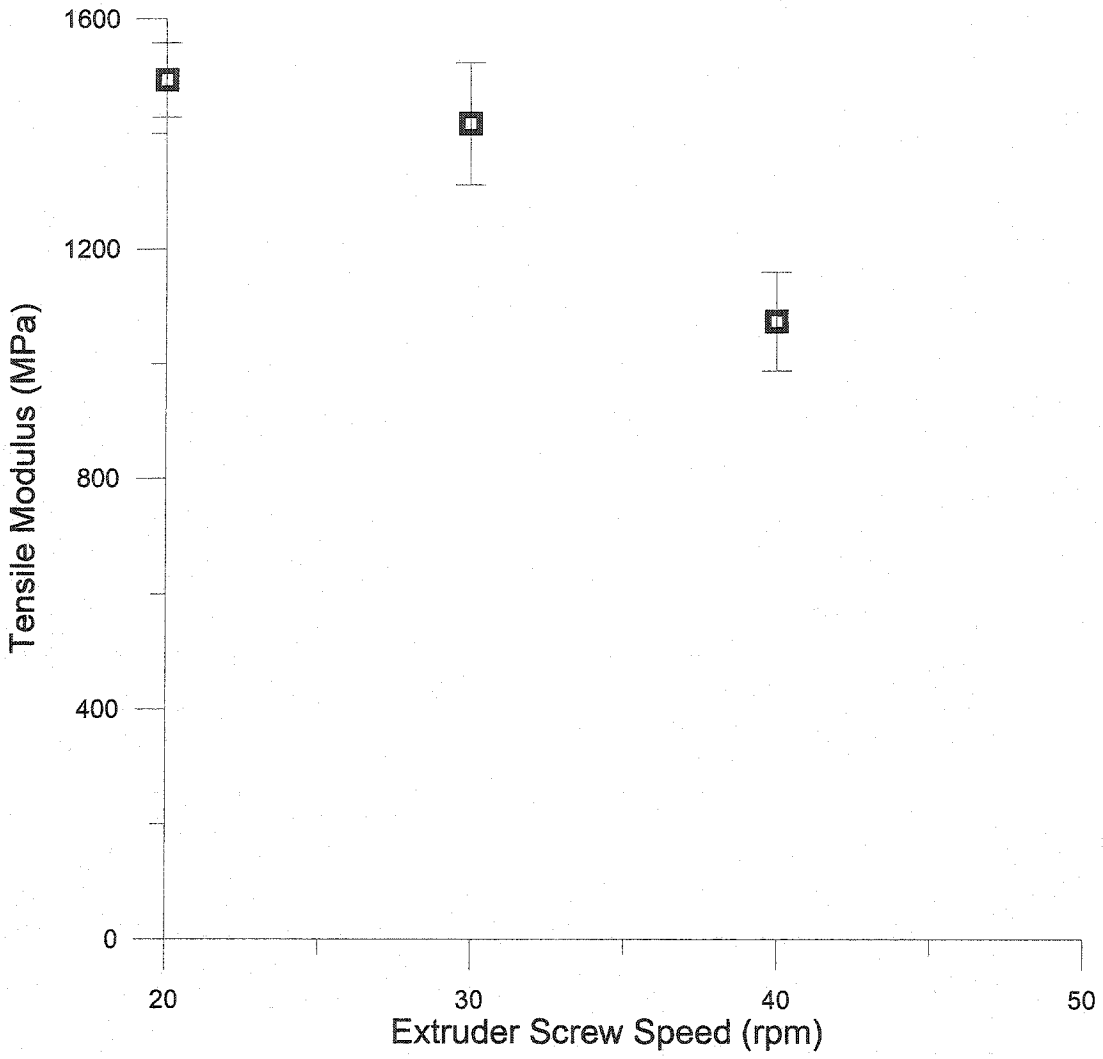


Figure (4.10) Tensile Modulus of palm/polypropylene composite with 30 wt% fiber vs extruder screw speed at Zone 1=195°C, Zone 2=200°C, Zone 3 =205°C, and Nozzle=210°C.

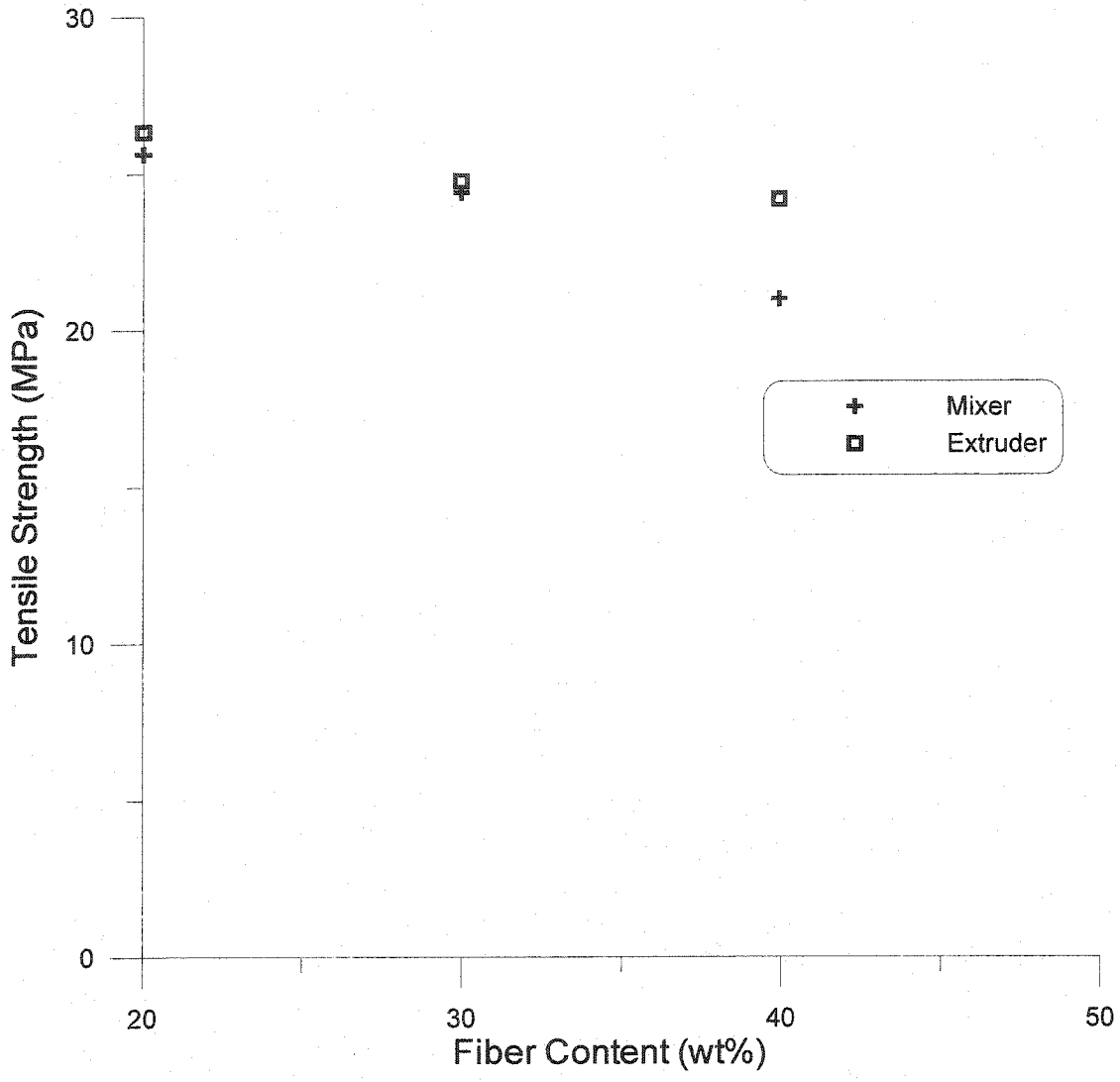


Figure (4.11) Tensile Strength of palm/polypropylene composite with 30 wt% fiber vs fiber content. Extruder conditions at Zone 1=195°C, Zone 2=200°C, Zone 3=205°C, and Nozzle 210°C. Mixer conditions: mixing time 8 min, rpm at 50 and temperature at 200°C.

4.2 Effects of Fiber (Size and Content) and Use of Compatibilizers on Composite Performance

Mechanical tests were performed on the composites formed to determine the effects of fiber size and utilization of compatibilizers Epolene E-43 and G 3003 on composite performance. The results obtained are discussed below.

Figure (4.12) shows the effect of fiber size and content on the tensile modulus of palm-polypropylene composite. As seen the increase in fiber content increases the composite modulus for both small and large fiber which is an indication for the existence of adhesion to some extent between polypropylene and much stiffer palm fiber.

Figure (4.13) shows the effect of fiber size and content on tensile strength of palm fiber-polypropylene composite. As the fiber content is increased the tensile strength decreases. This might be attributed to the poor adhesion between the fiber and polypropylene. So, does this result contradict the improvement in composite stiffness with fiber addition as just discussed above? The answer is no. The modulus is related to the stiffness of the material before fracture and it is obtained from the slope of the straight portion of the stress-strain curve. Existence of adhesion (even weak) between fibers and the matrix would then improve composite modulus. However, composite tensile strength is a result of material fracture at the weakest point of the material, which might occur below the matrix (polypropylene) strength if the adhesion between the fibers and the matrix fails at a lower stress.

Figure (4.14) shows the effect of fiber loading on the tensile elongation of palm-polypropylene composite. We can see that the tensile elongation drops considerably with 20 wt% fiber addition and keeps dropping with the increase in fiber content, it is a common observation with almost all filled composites where elongation decreases with addition of more fiber to the polymer. The ductility of polypropylene has been suppressed by the presence of fibers. The reduction may be attributed to the decreased deformability of a rigid interface between the fiber and the matrix material. At higher fiber loading, the domination of fiber matrix interaction can be expected to diminish and being replaced with filler-filler interaction [Rozman et al., 2000].

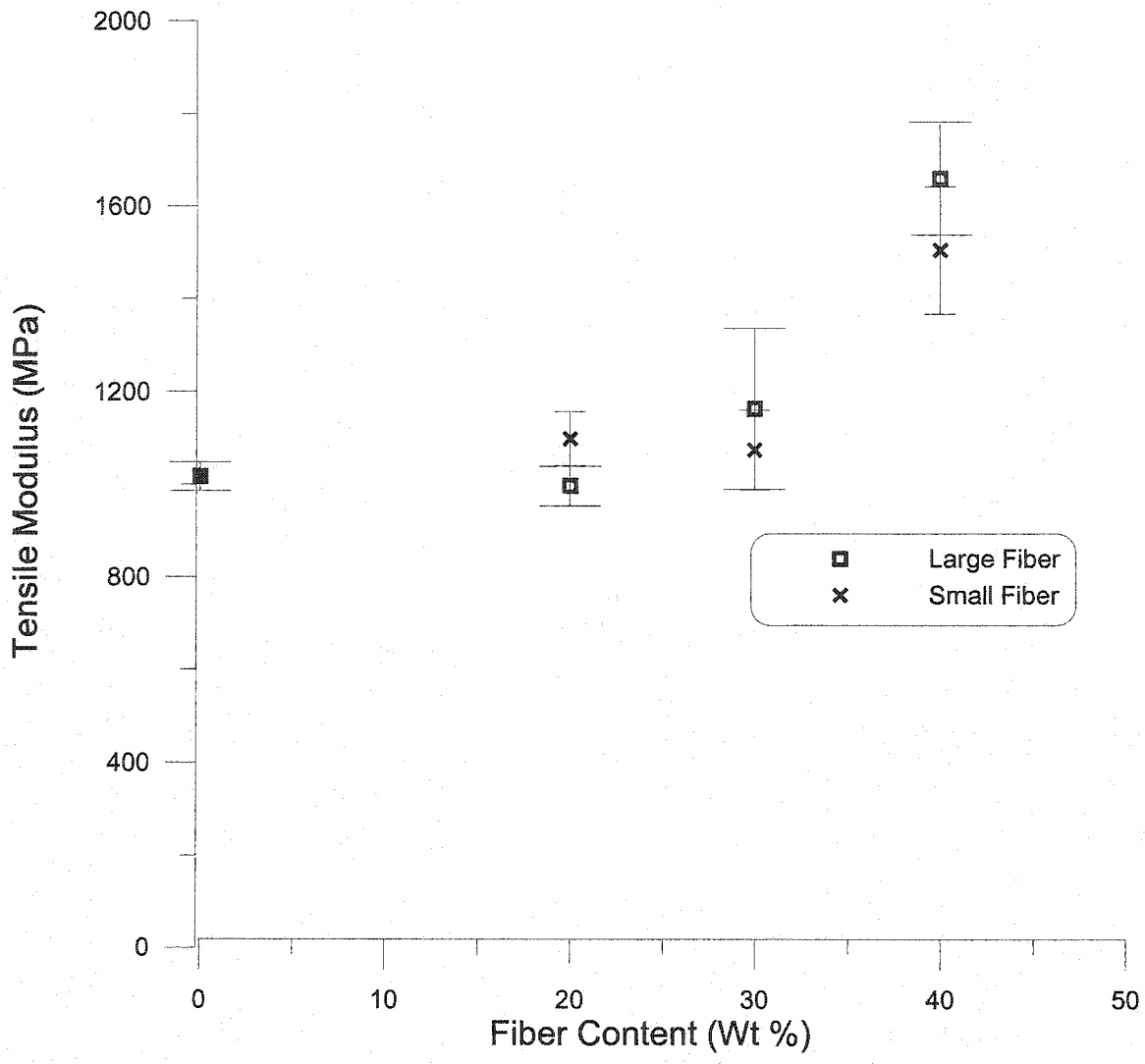


Figure (4.12) Tensile modulus vs fiber content for polypropylene reinforced with small and large palm fiber.

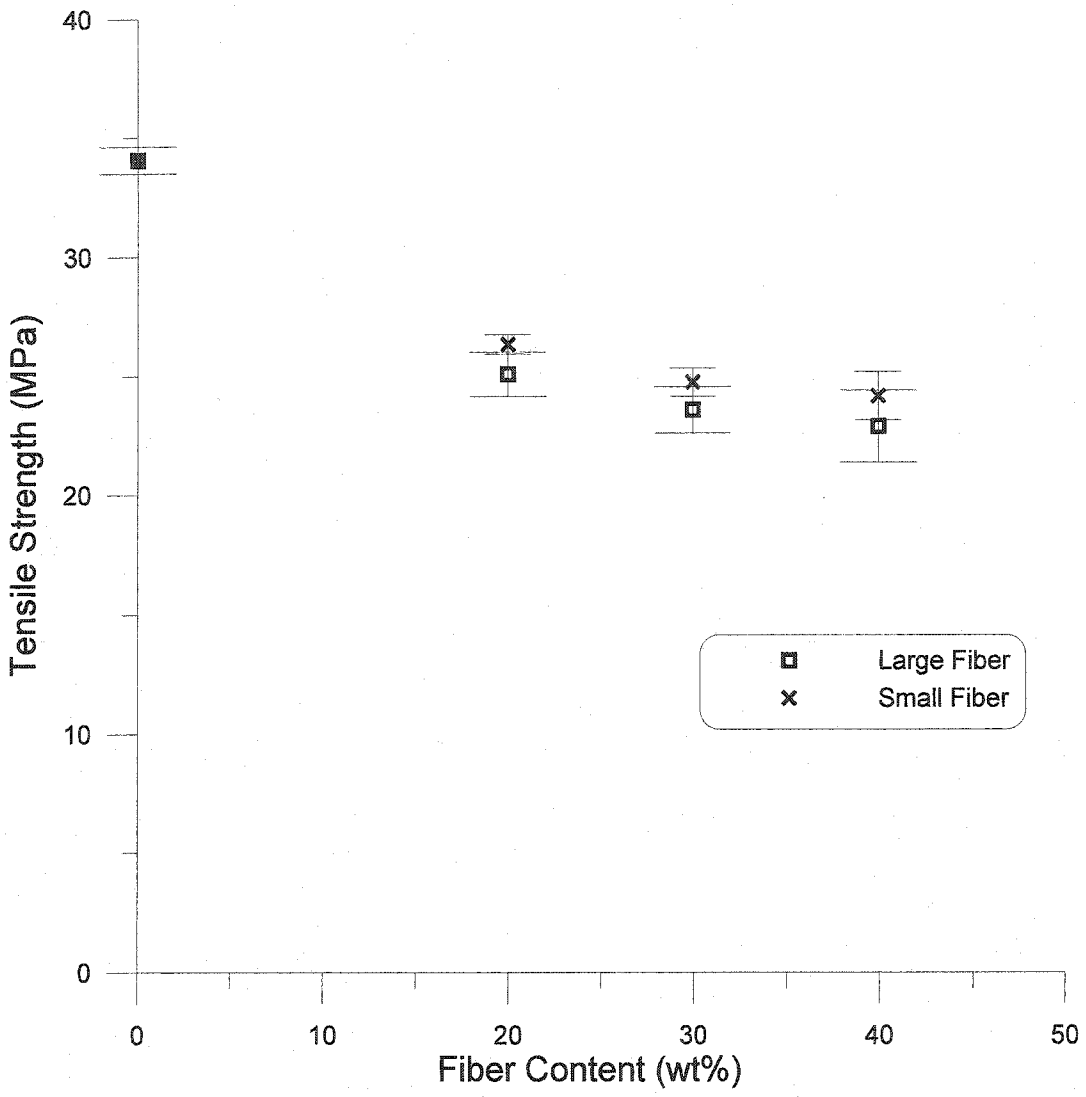
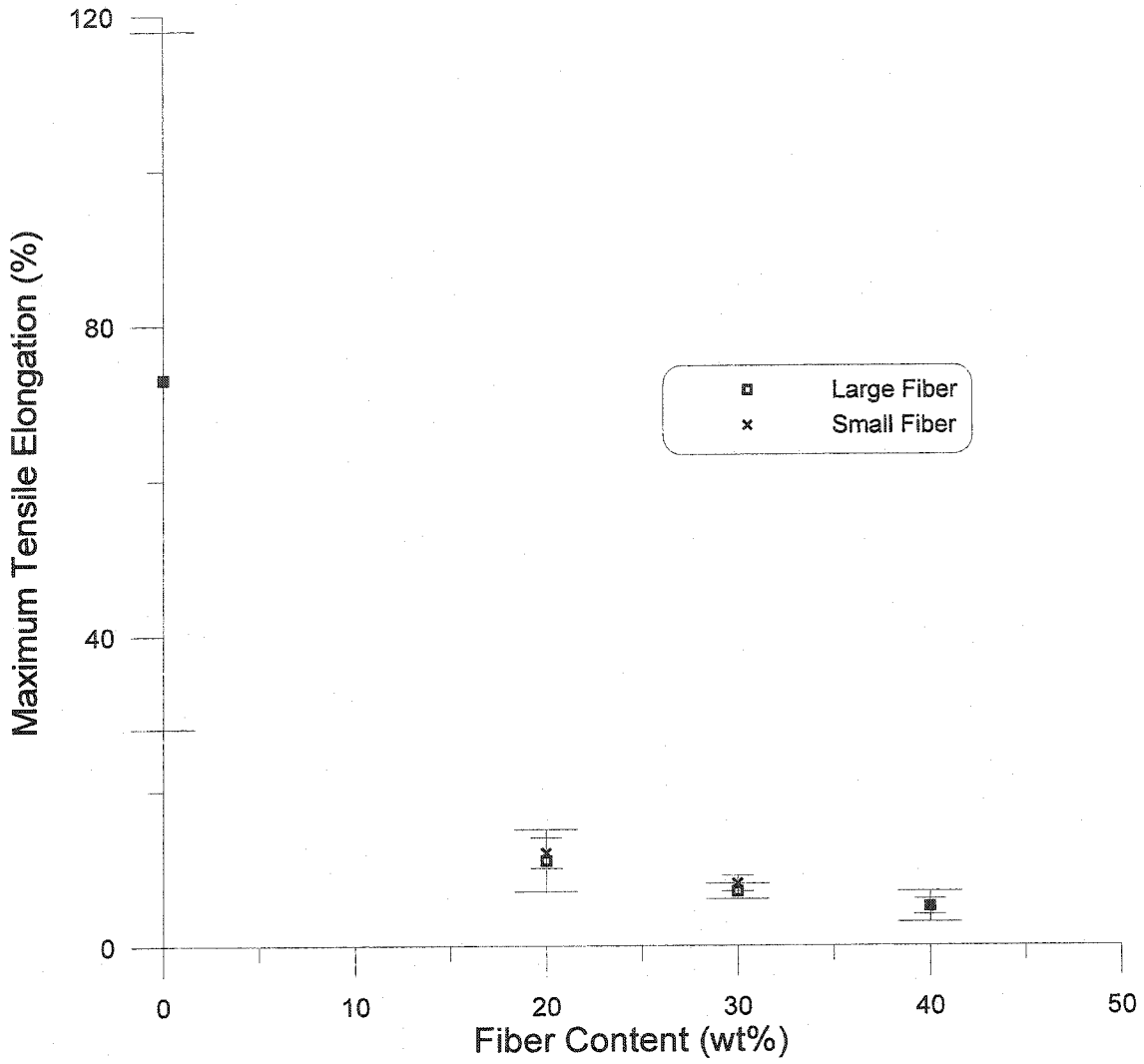


Figure (4.13) Tensile strength vs fiber content for polypropylene reinforced with small and large palm fiber.



Figure(4.14) Maximum tensile elongation vs fiber content for polypropylene reinforced with small and large palm fiber.

As also shown in Figure (4.12) and (4.13), the difference in the results obtained with small or large fibers was not significant considering also the scatter in data. But still the composite strength with small fiber was observed to be consistently higher than that with large fibers while the same effect was not observed in the case of composite modulus. This was probably because of better mixing and more uniform distribution of small fibers in the polypropylene matrix as compared to large fibers. Accumulation or non uniform orientation of fibers at some parts of the composite can result in fracture at these weak points giving lower strength in the case of larger fibers. Non uniformities in fiber distribution here and there (if not so extensive) would not affect the composite modulus at the same extent since the modulus is the bulk material property before fracture.

The next step was to utilize compatibilizers to see their effect on composite performance. Figures (4.15) and (4.16) show the effect of adding 2 wt% Epolene E-43 on the tensile strength and modulus of the composite respectively, with varying fiber loading and fiber length distribution. The composite behavior is similar to that of the uncompatibilized one. Again the tensile strength drops as fiber loading is increased. Small fibers result in somewhat better composite strength than large fibers, and the modulus increases with the increasing fiber loading. However, higher composite strength values relative to that of uncompatibilized ones is an indication of some improvement in fiber-matrix adhesion with 2 wt% Epolene E-43.

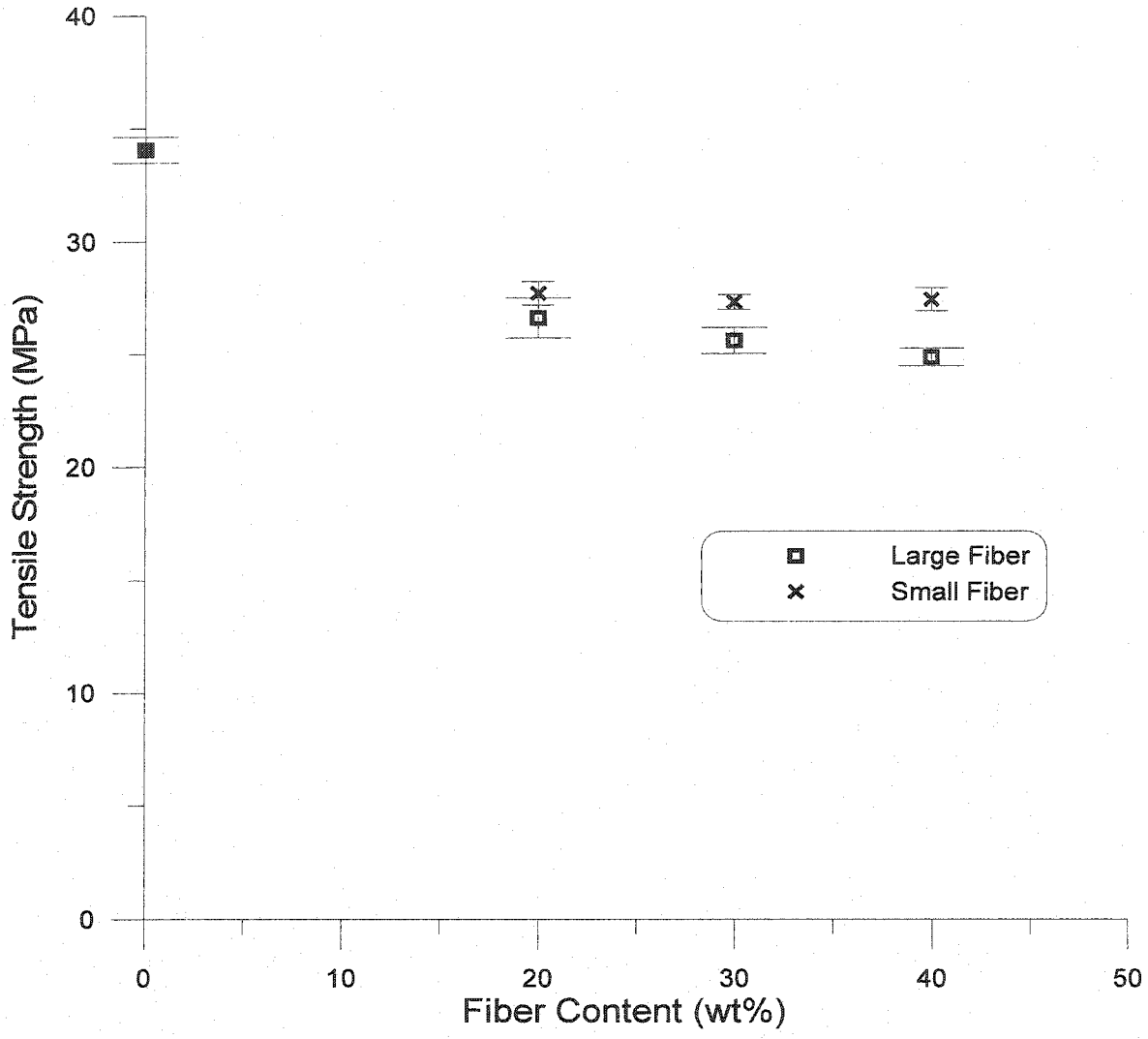


Figure (4.15) Tensile strength vs fiber content for palm fiber-polypropylene matrix composites compatibilized by 2 wt% Epolene E-43.

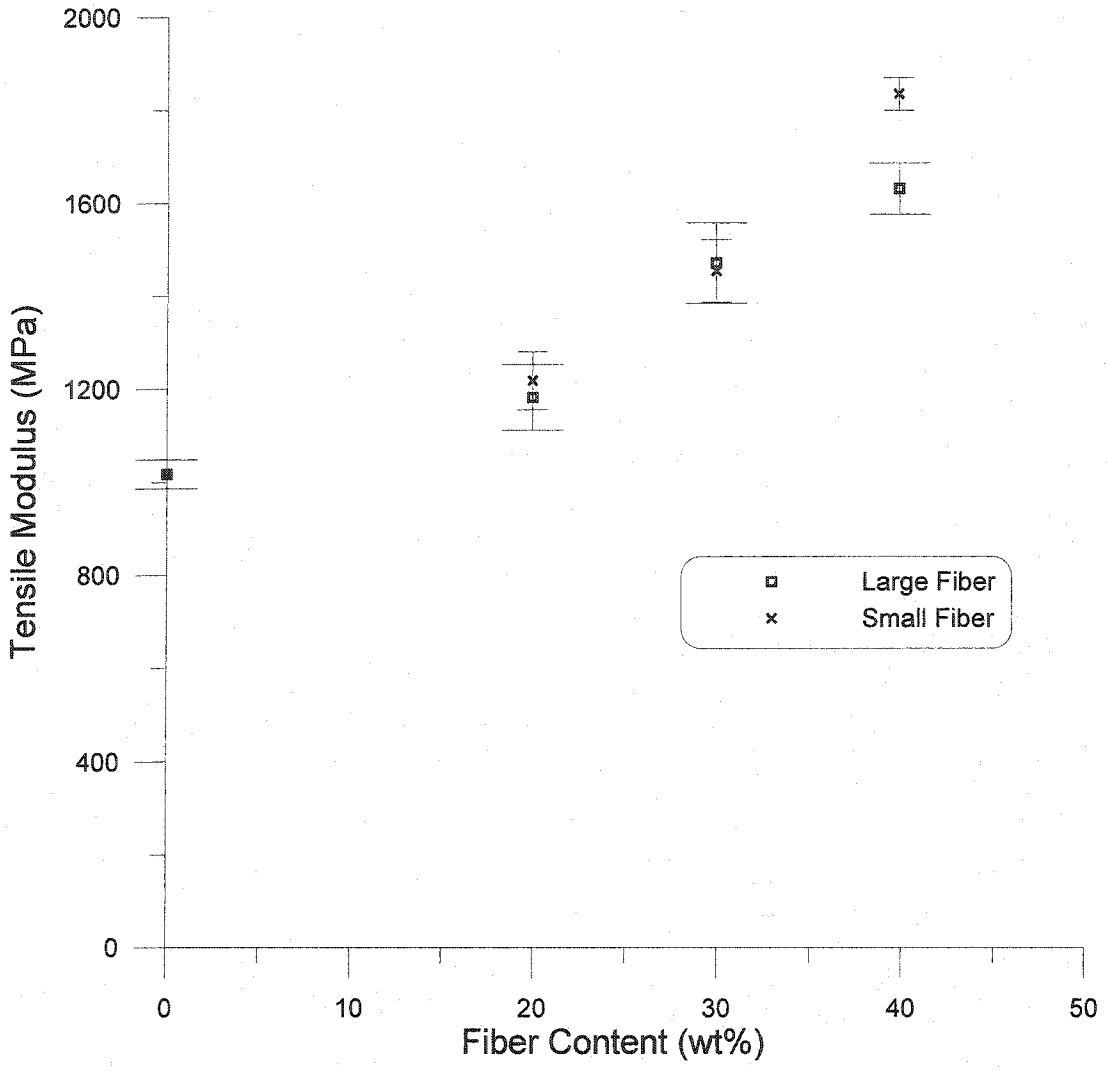


Figure (4.16) Tensile Modulus vs fiber content for palm fiber-polypropylene matrix composites compatibilized by 2 wt% Epolene E-43.

Figure (4.17) shows the effect of compatibilizer Epolene E-43 on tensile elongation with varying fiber loading and fiber length distribution. It is seen that the adding 2% Epolene E-43 does not improve the tensile elongation.

Figures (4.18) and (4.19) show the effect of 4 wt% Epolene E-43 on composite strength and modulus with varying fiber loading and fiber length distribution. The composite behavior is similar to that of the composite compatibilized with 2wt% Epolene E-43 just discussed above.

Figure (4.20) shows the effect of (4% by weight) compatibilizer Epolene E-43 on tensile elongation with varying fiber loading and fiber length distribution. Addition of 4% Epolene E-43 didn't impart an improvement to the tensile elongation.

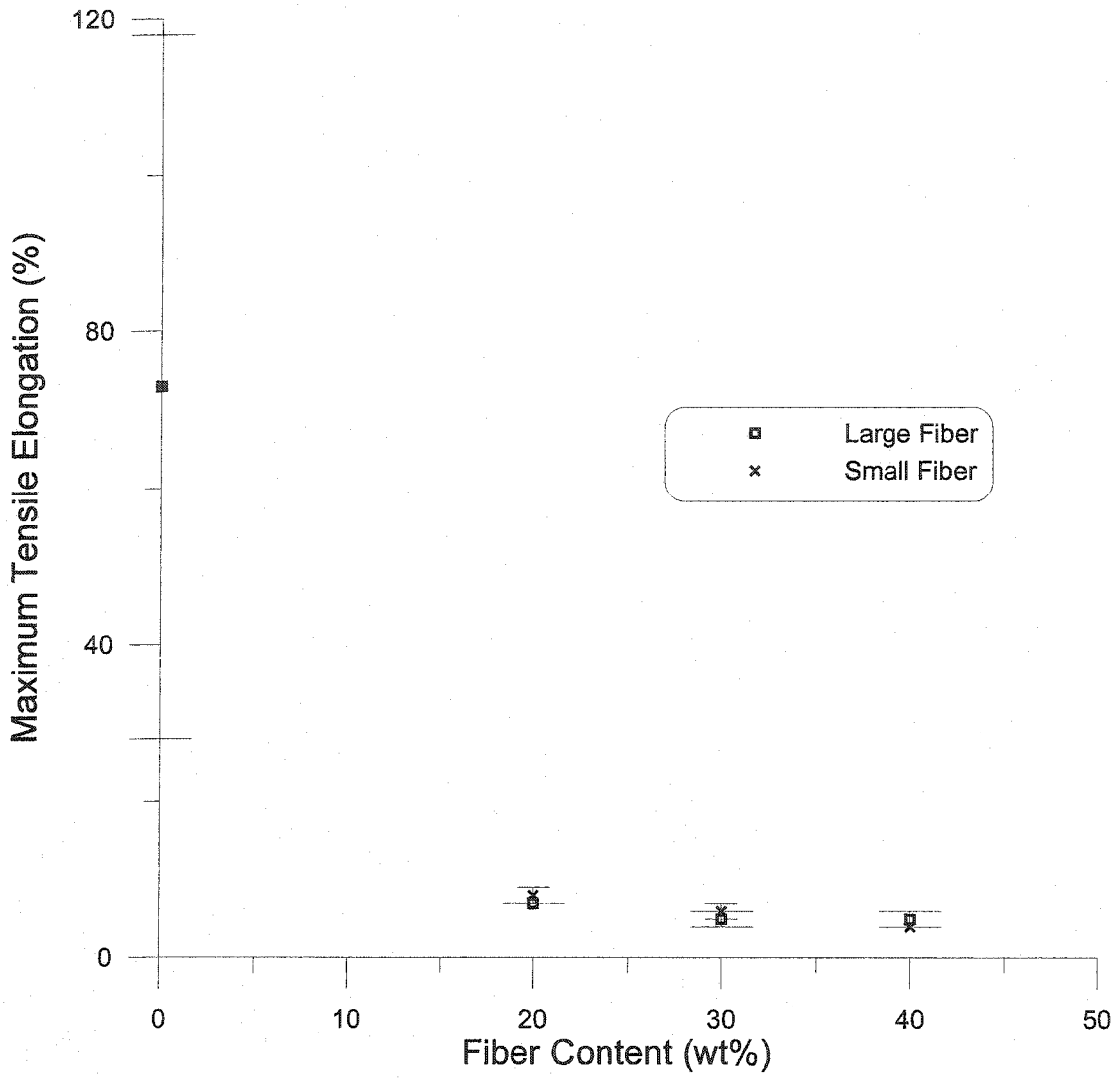


Figure (4.17) Tensile Elongation vs fiber content for palm fiber-polypropylene matrix composites compatibilized by 2 wt% Epolene E-43.

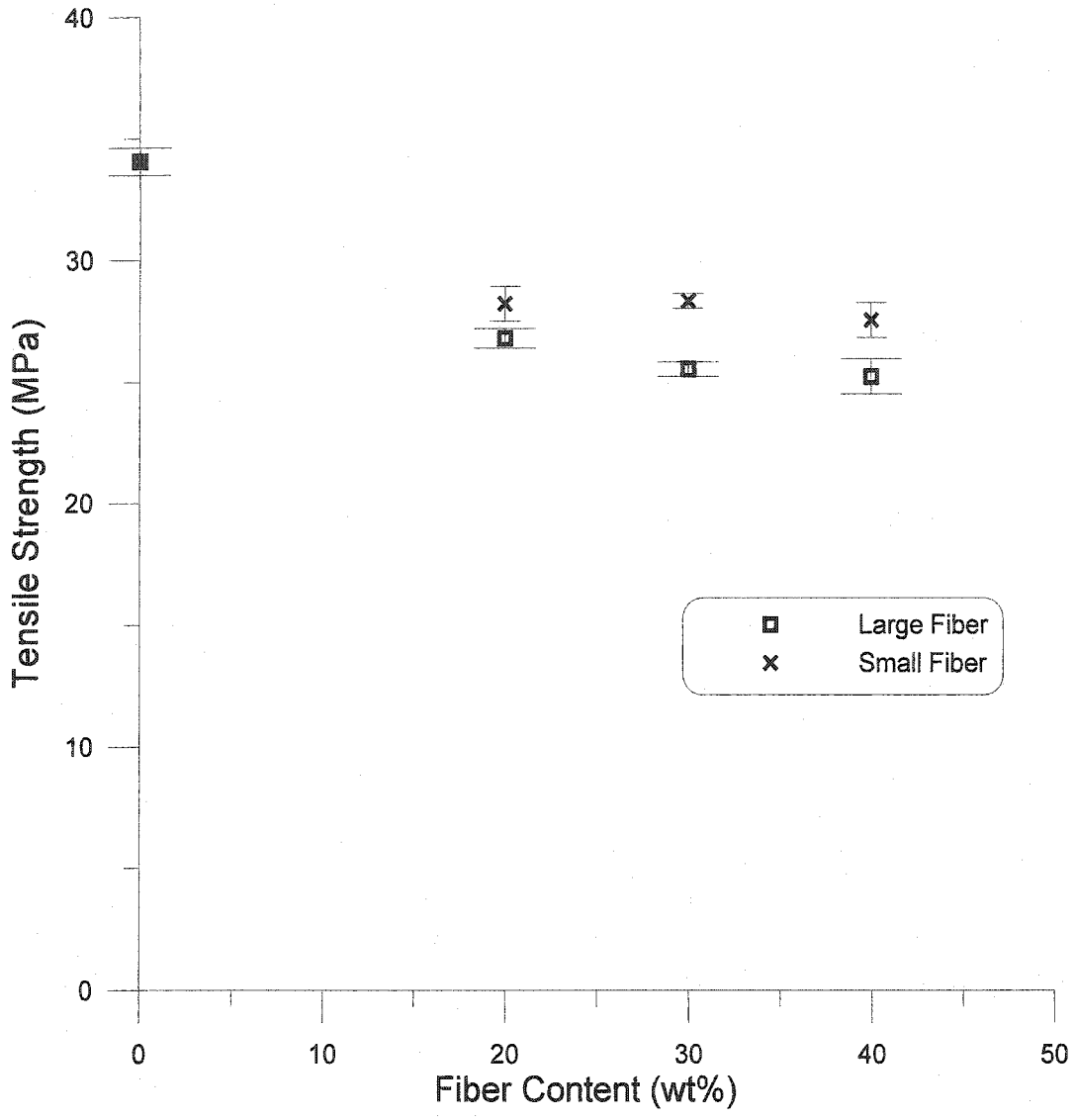


Figure (4.18) Tensile strength vs fiber content for palm fiber-polypropylene matrix composites compatibilized by 4 wt% Epolene E-43.

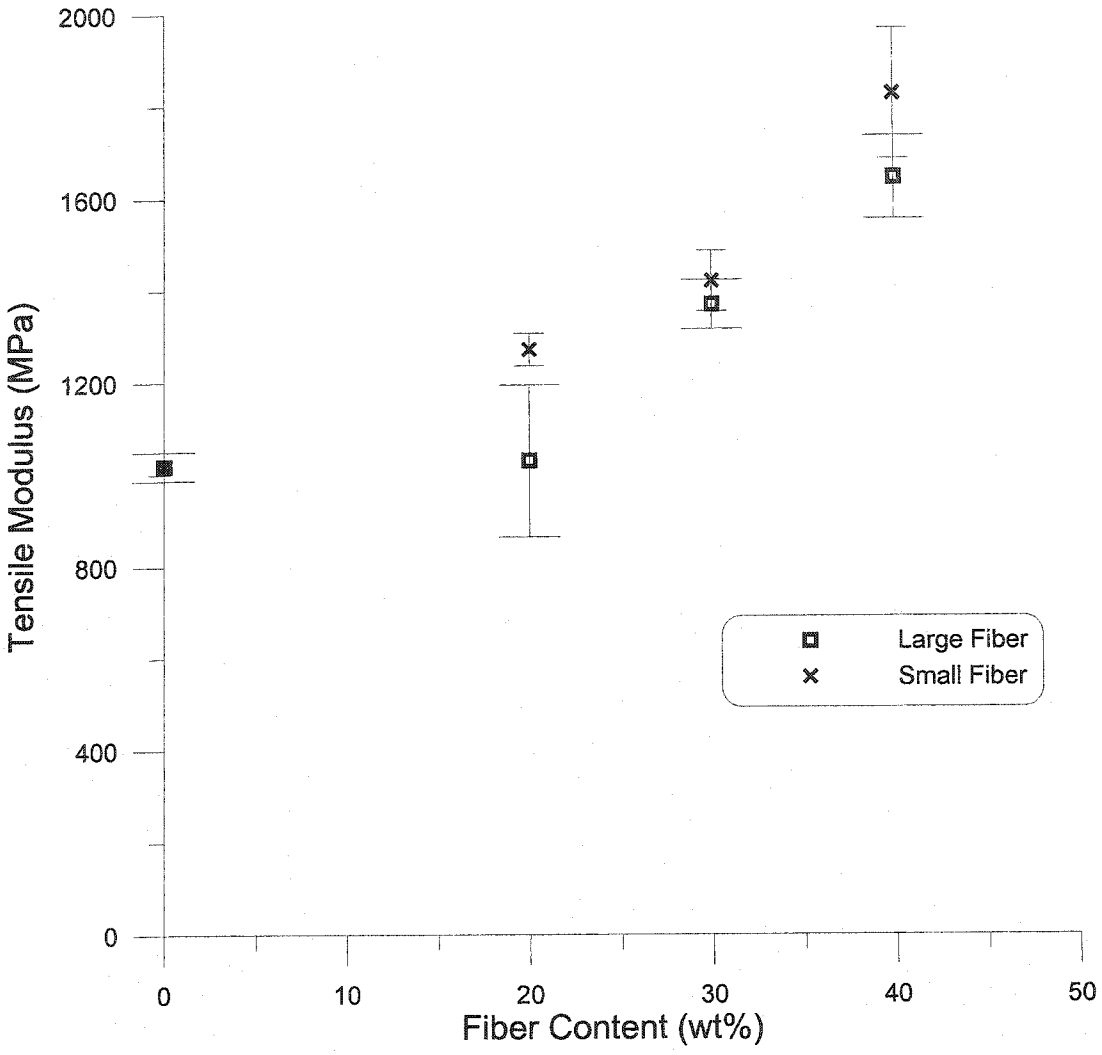


Figure (4.19) Tensile modulus vs fiber content for palm fiber-polypropylene matrix composites compatibilized by 4 wt% Epolene E-43.

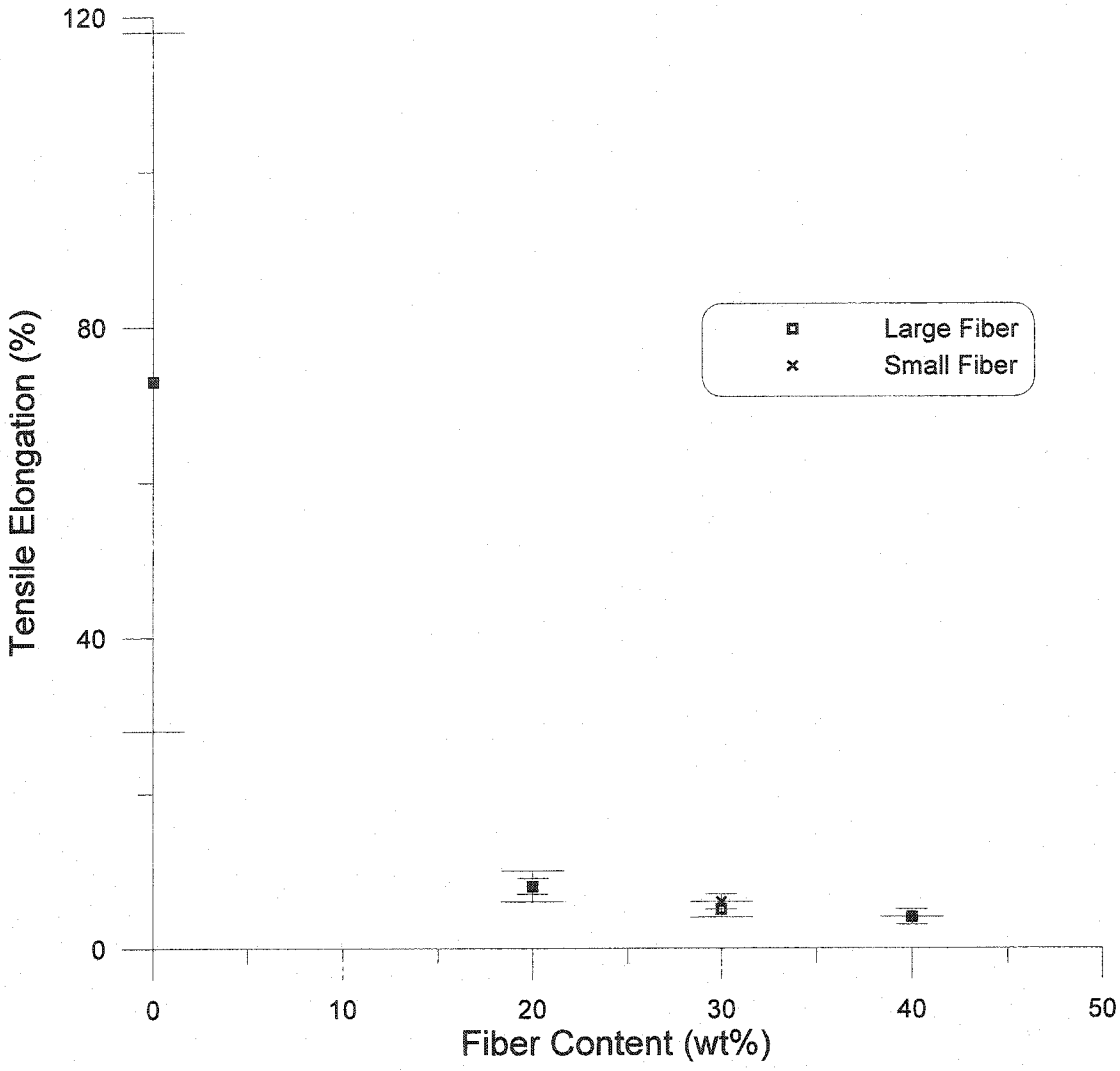


Figure (4.20) Tensile elongation vs fiber content for palm fiber-polypropylene matrix composites compatibilized by 4 wt% Epolene E-43.

The performance of the composite with 6 wt% compatibilizer Epolene E-43 was not different either as shown in Figures (4.21), (4.22) and (4.23).

Mechanical performance of the composite improved further with utilization of compatibilizer Epolene G-3003 [Figures (4.24) – (4.32)] and the composite strength reached close to that of unreinforced polypropylene with 6 wt% Epolene G-3003 [Figure (4.30)].

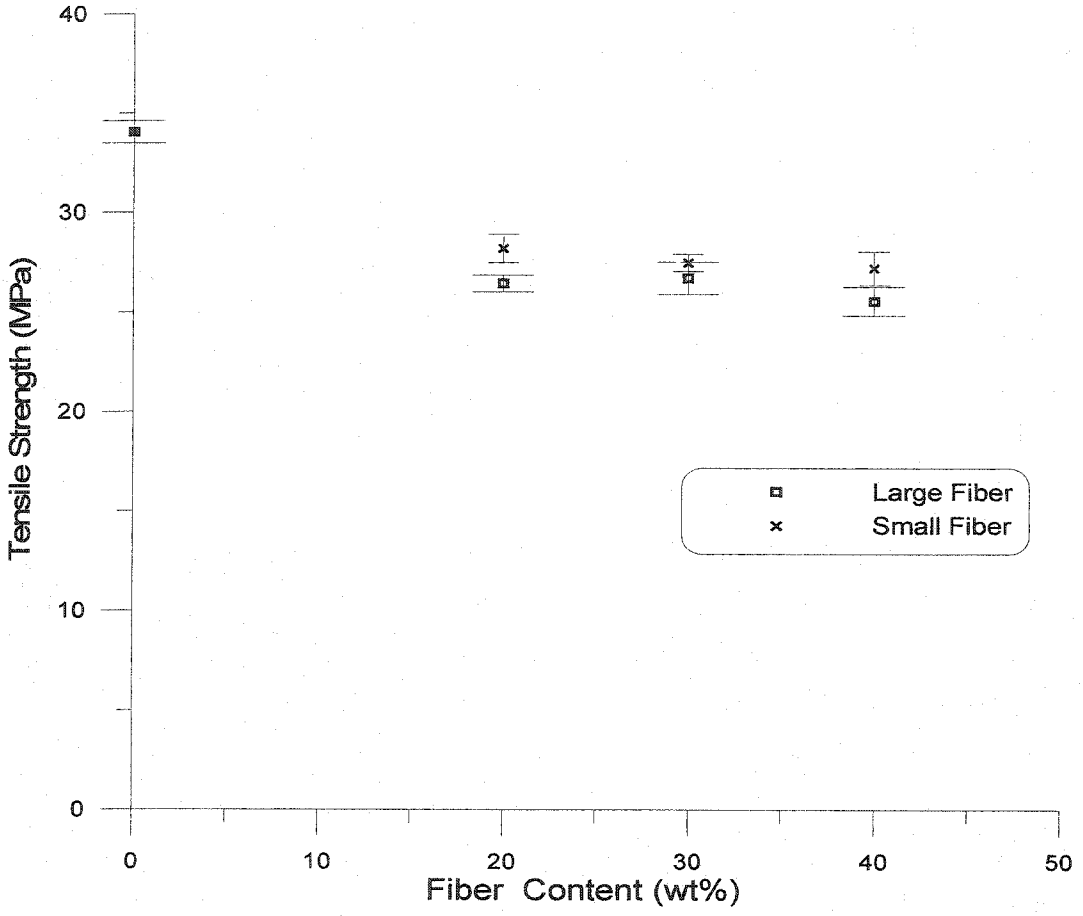


Figure (4.21) Tensile strength vs fiber content for palm fiber-polypropylene matrix composites compatibilized by 6 wt% Epolene E-43.

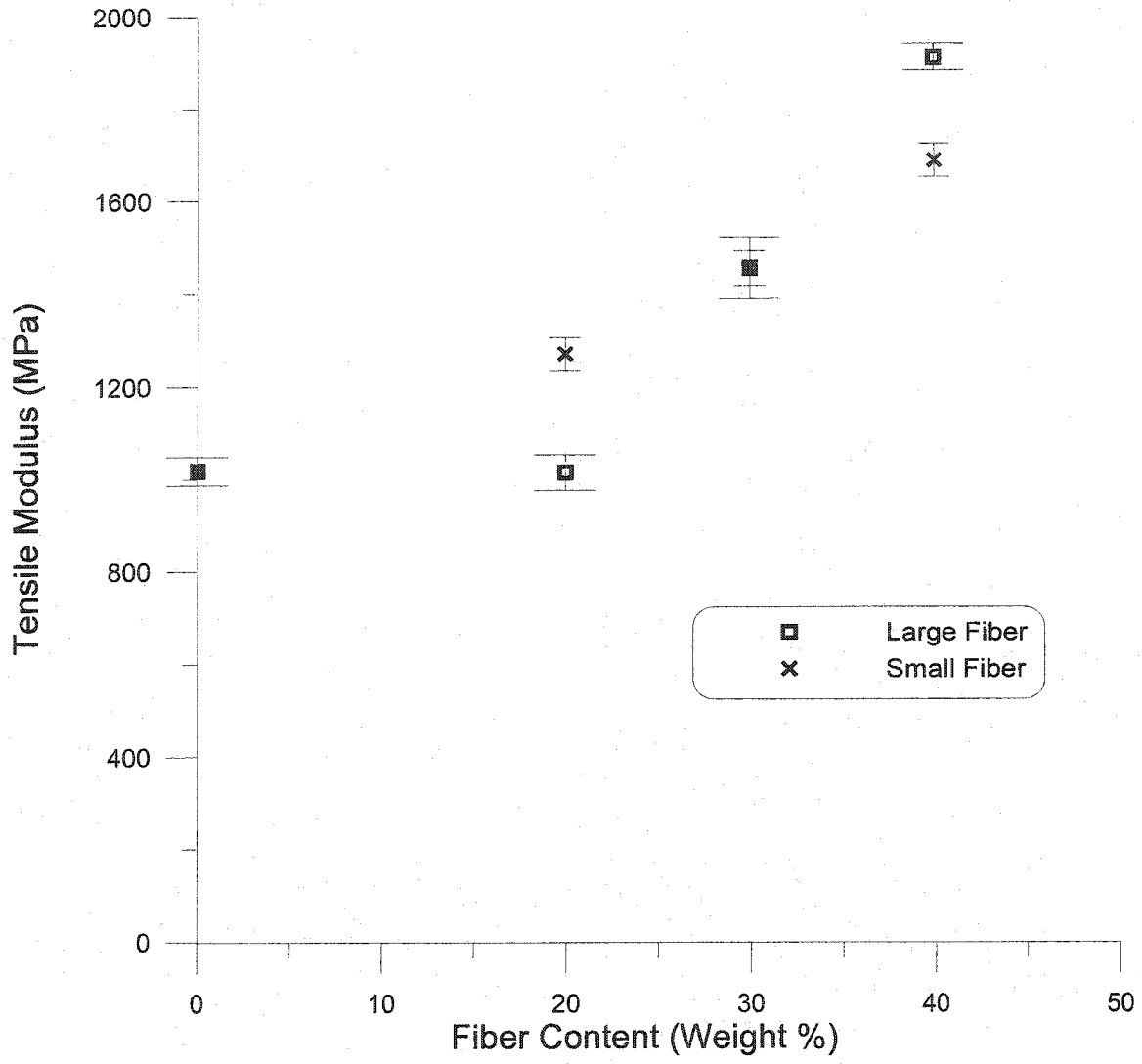


Figure (4.22) Tensile modulus vs fiber content for palm fiber-polypropylene matrix composites compatibilized by 6 wt% Epolene E-43.

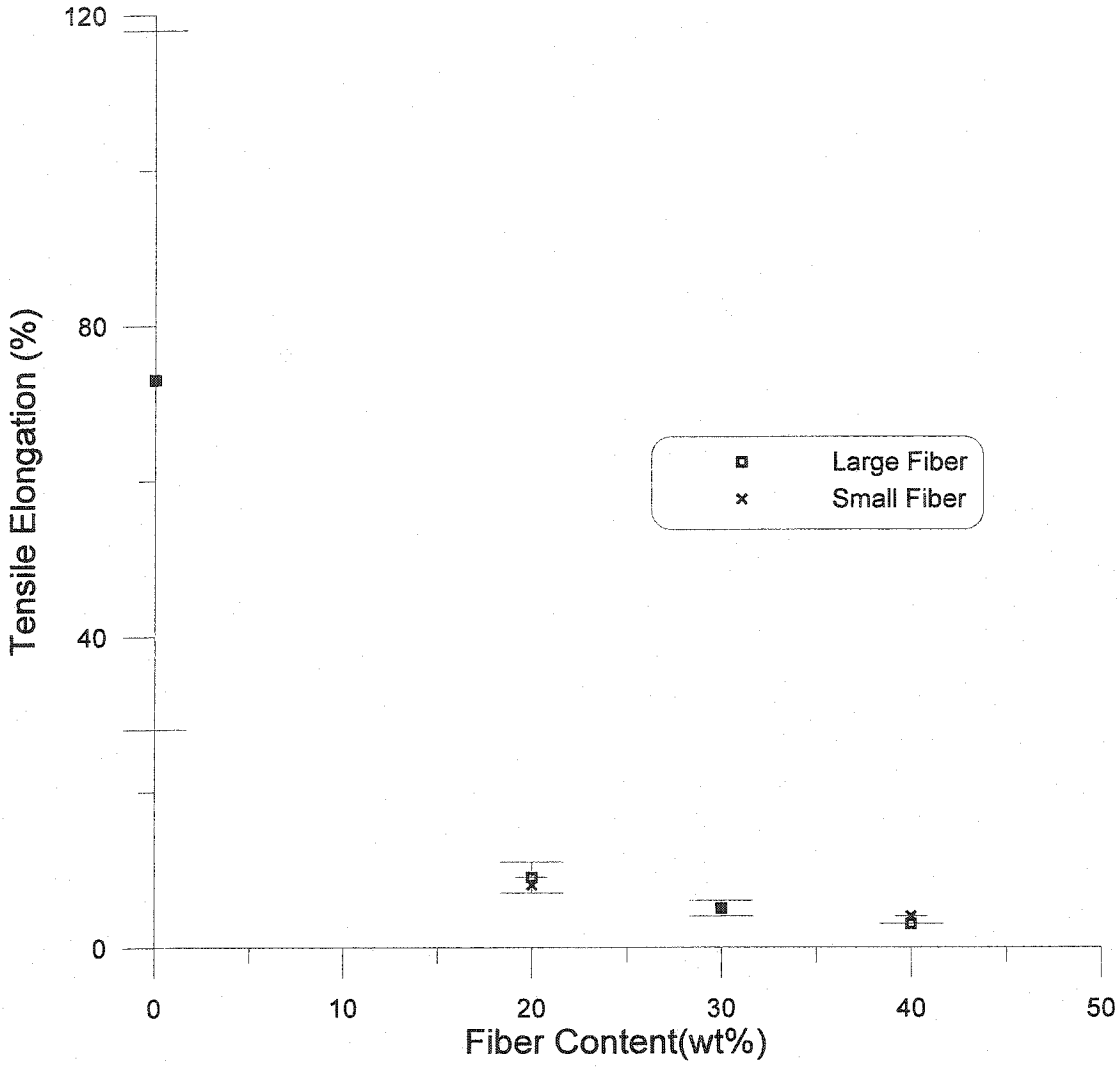


Figure (4.23) Tensile elongation vs fiber content for palm fiber-polypropylene matrix composites compatibilized by 6 wt% Epolene E-43.

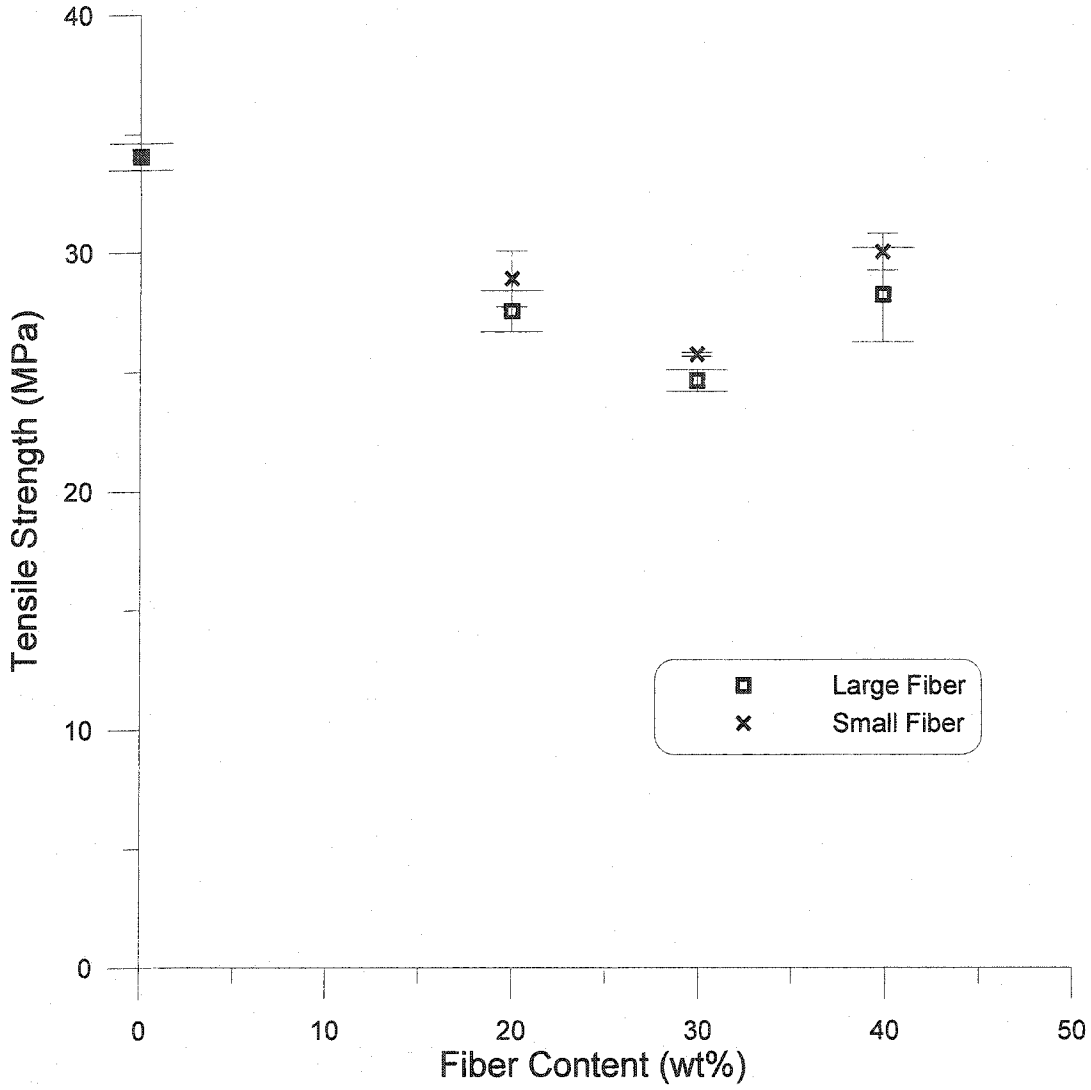


Figure (4.24) Tensile strength vs fiber content for palm fiber-polypropylene matrix composites compatibilized by 2 wt% Epolene G-3003.

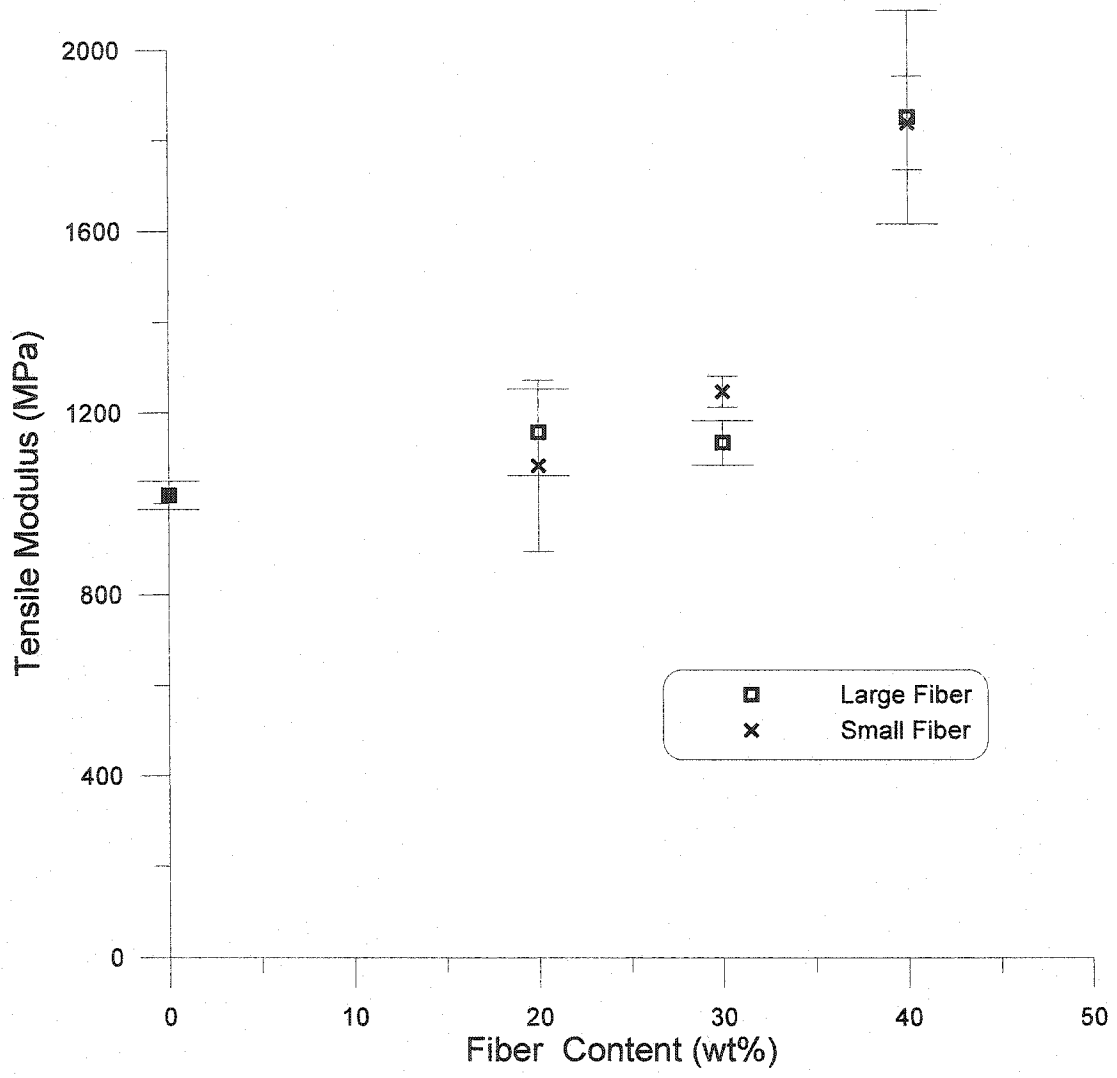


Figure (4.25) Tensile modulus vs fiber content for palm fiber-polypropylene matrix composites compatibilized by 2 wt% Epolene G-3003.

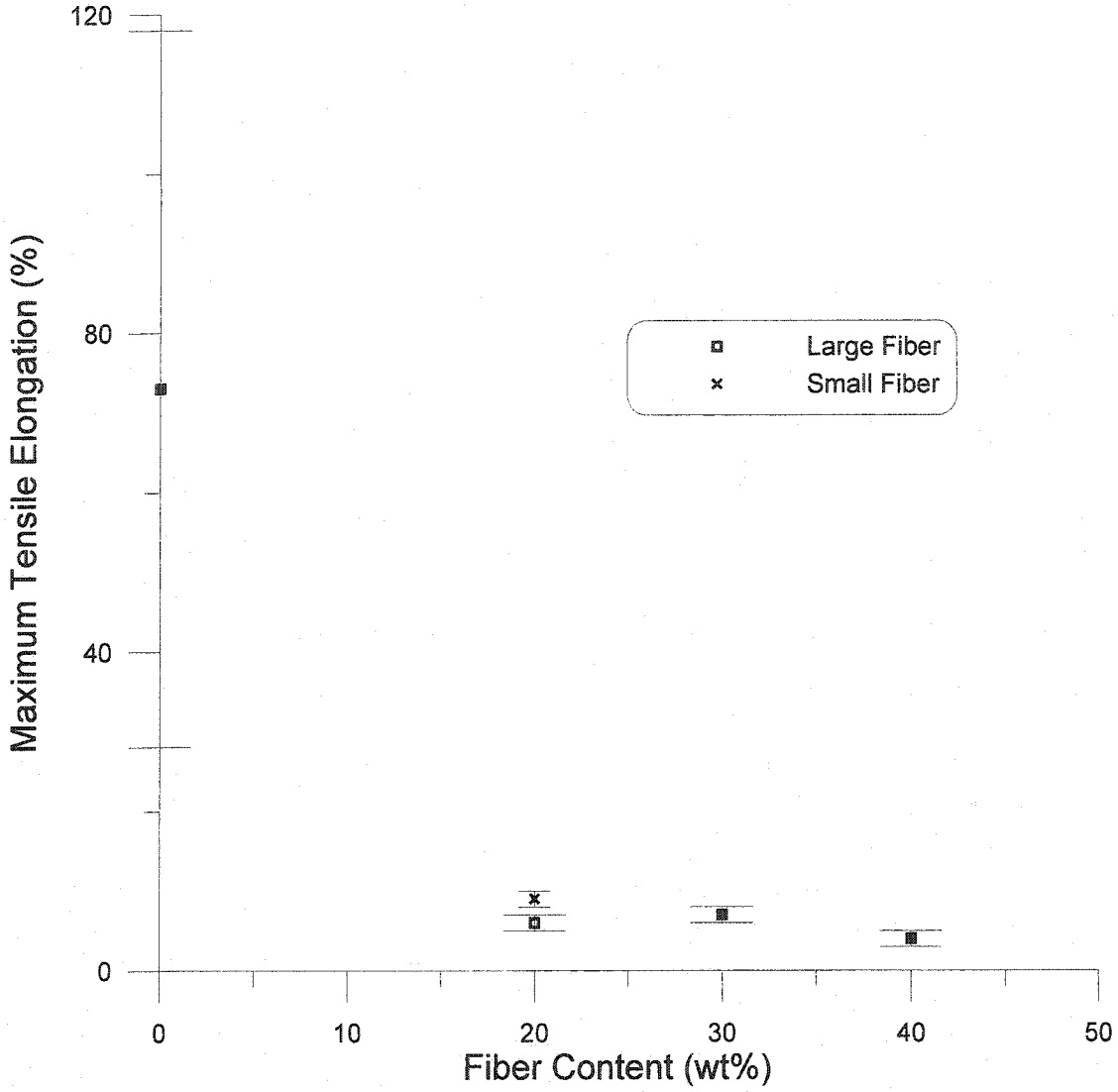


Figure (4.26) Tensile elongation vs fiber content for palm fiber-polypropylene matrix composites compatibilized by 2 wt% Epolene G-3003.

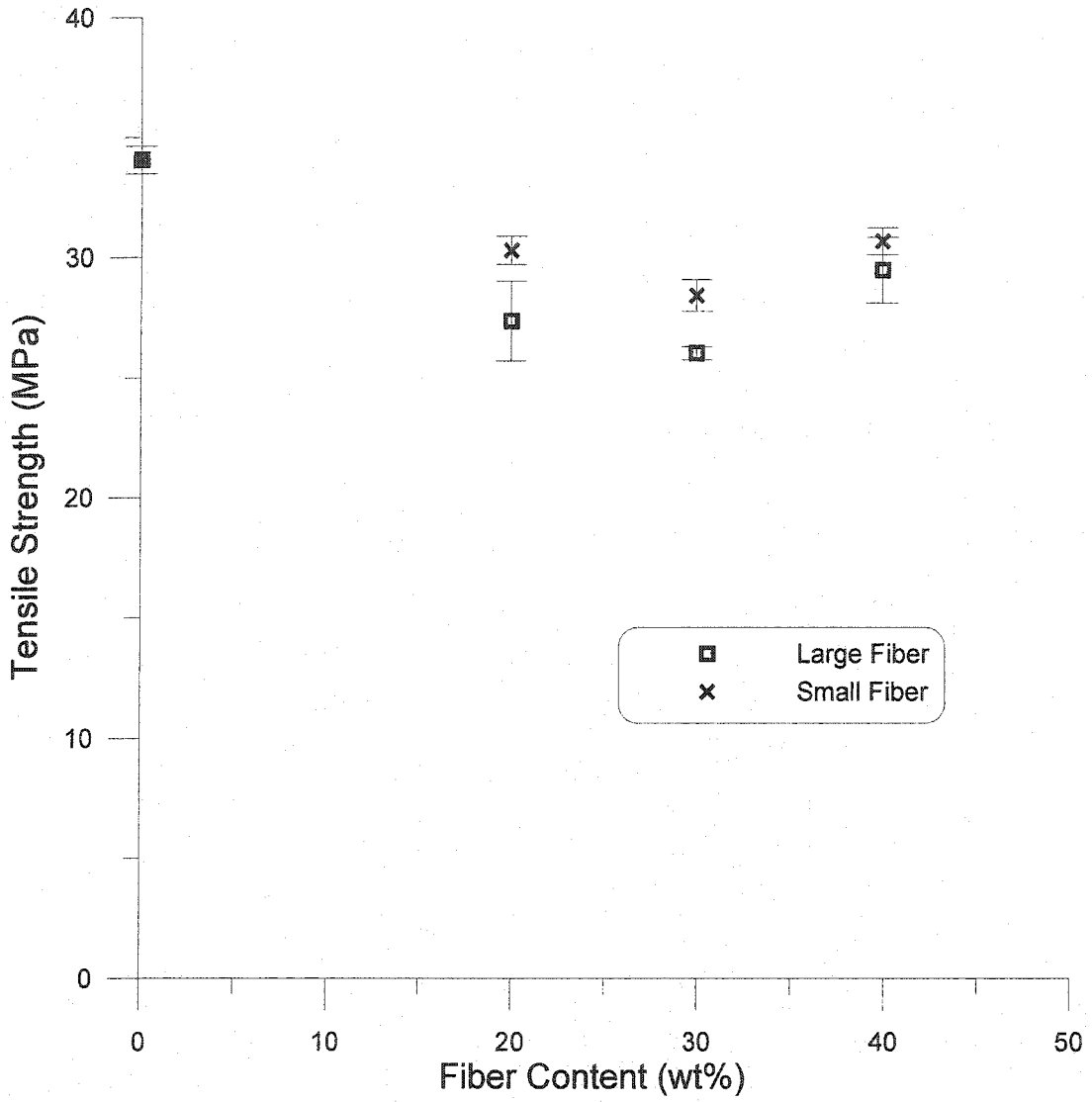


Figure (4.27) Tensile strength vs fiber content for palm fiber-polypropylene matrix composites compatibilized by 4 wt% Epolene G-3003.

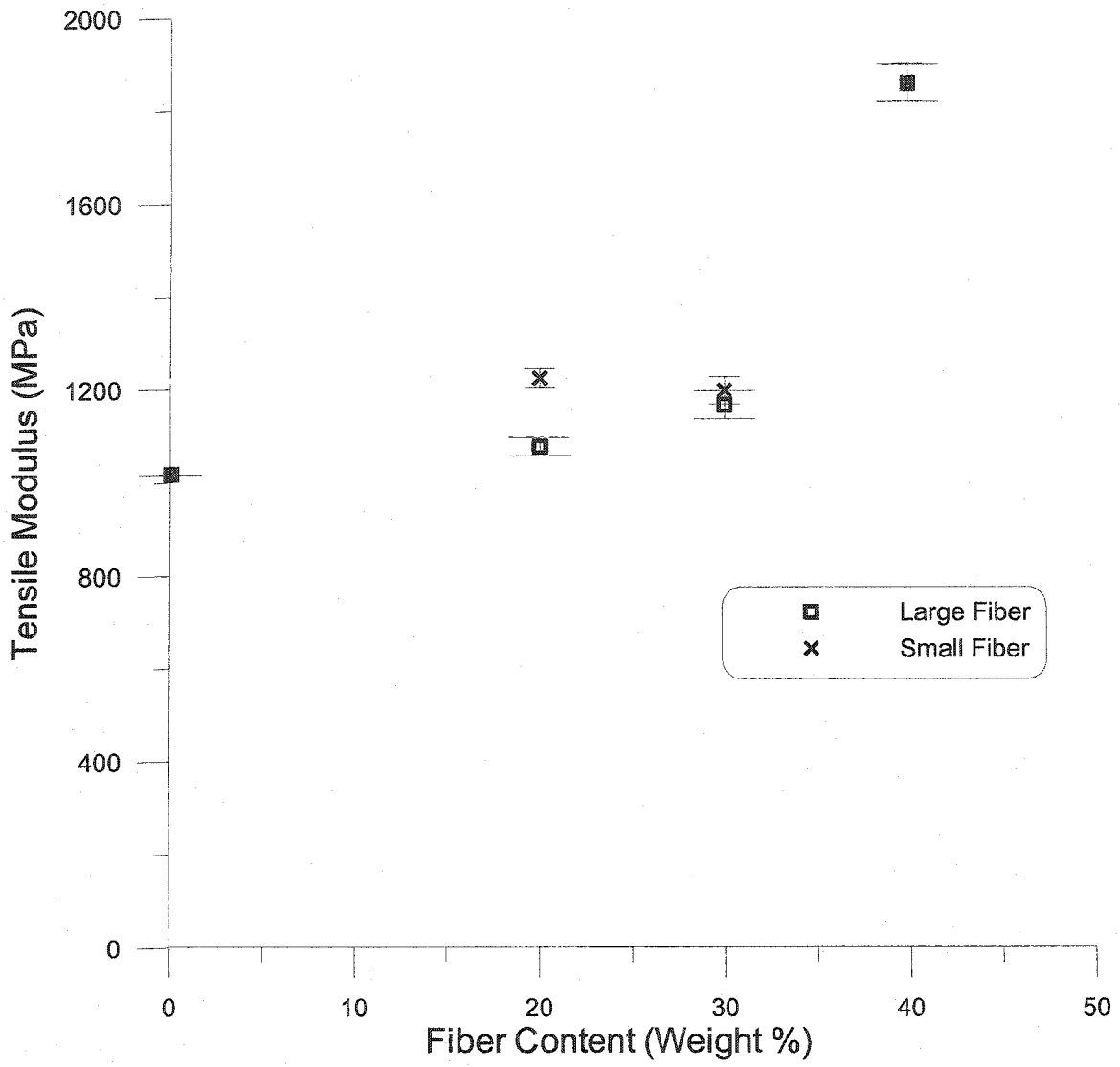


Figure (4.28) Tensile modulus vs fiber content for palm fiber-polypropylene matrix composites compatibilized by 4 wt% Epolene G-3003.

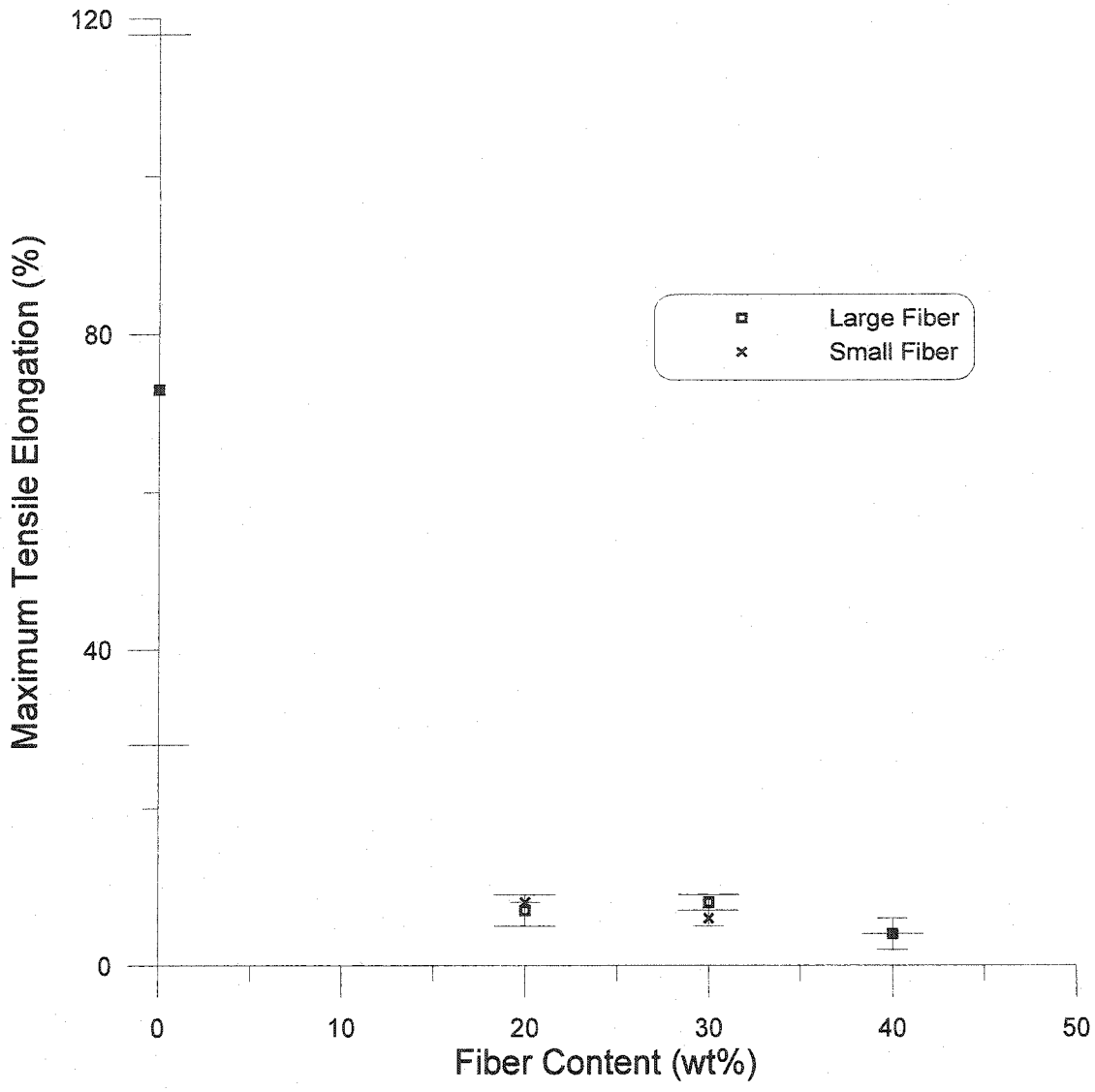


Figure (4.29) Tensile elongation vs fiber content for palm fiber-polypropylene matrix composites compatibilized by 4 wt% Epolene G-3003.

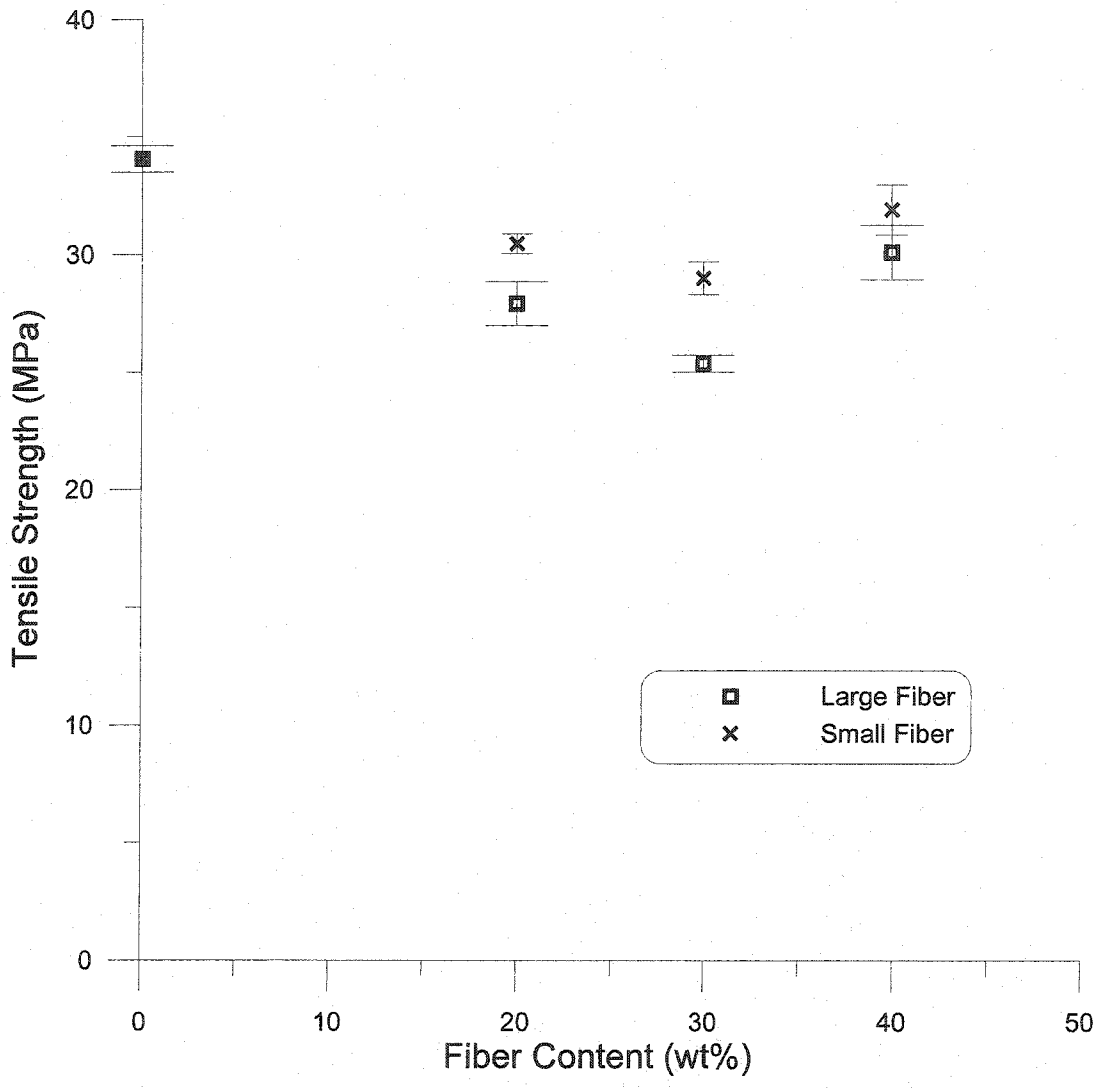


Figure (4.30) Tensile strength vs fiber content for palm fiber-polypropylene matrix composites compatibilized by 6 wt% Epolene G-3003.

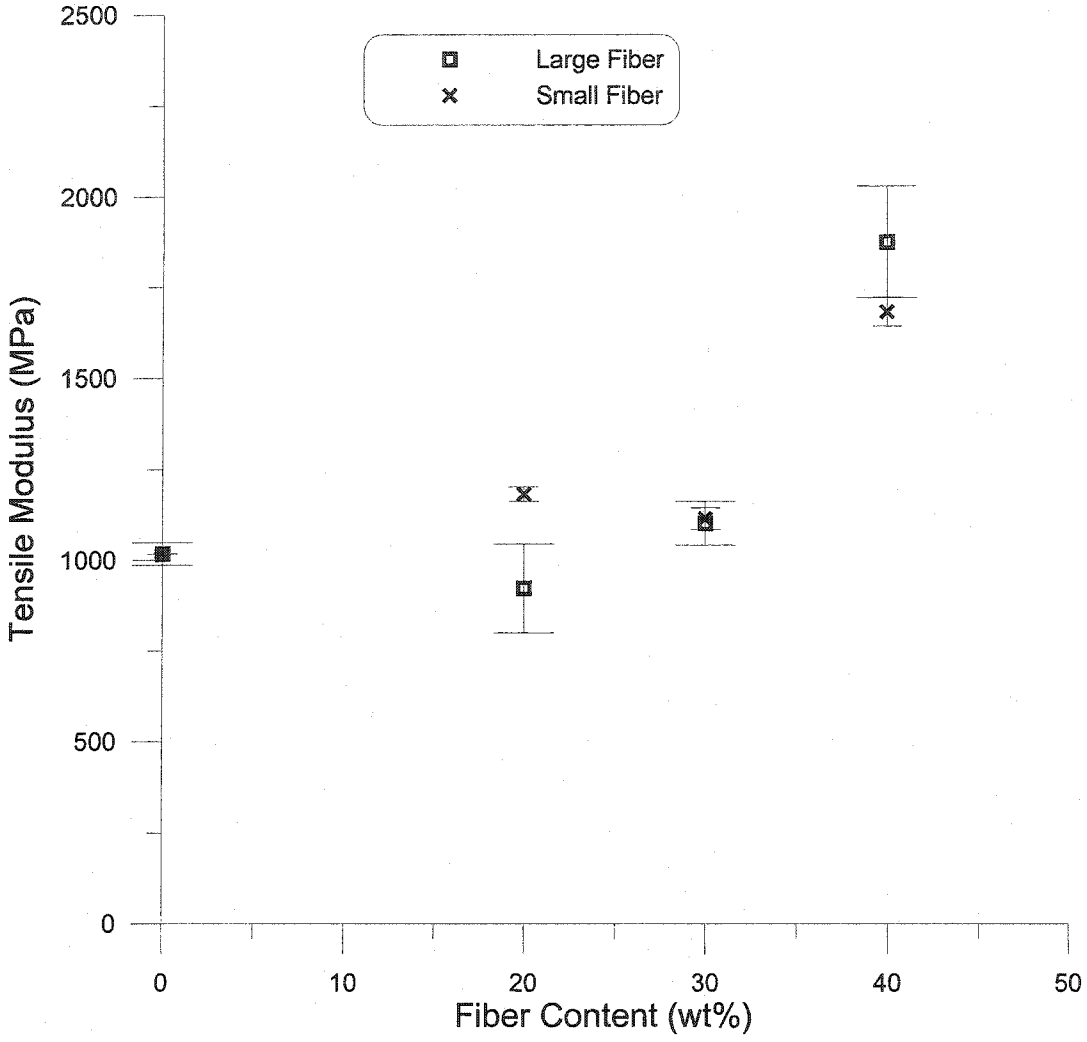


Figure (4.31) Tensile modulus vs fiber content for palm fiber-polypropylene matrix composites compatibilized by 6 wt% Epolene G-3003.

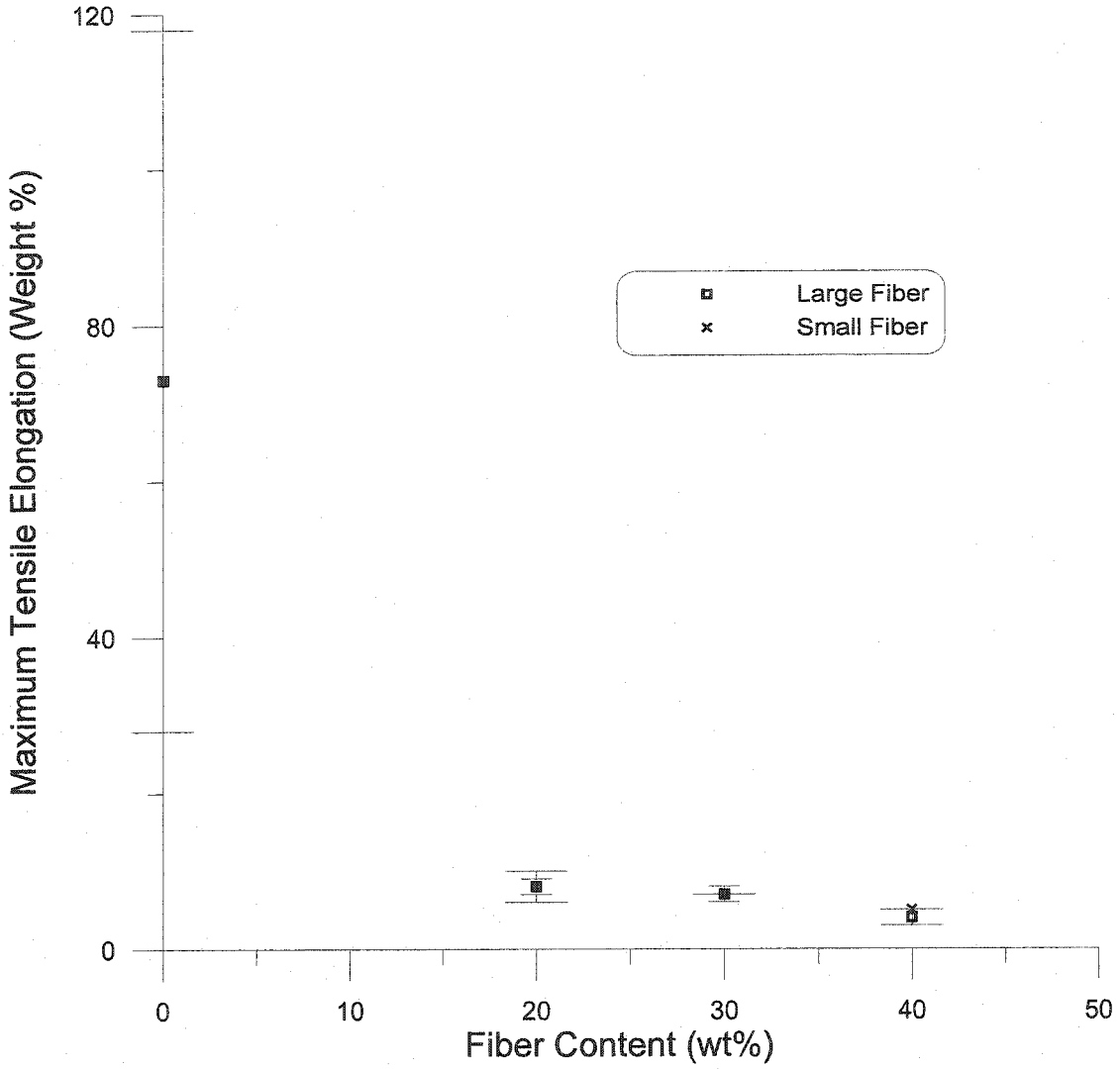


Figure (4.32) Tensile elongation vs fiber content for palm fiber-polypropylene matrix composites compatibilized by 6 wt% Epolene G-3003.

The results discussed above were combined together in Figures (4.33)-(4.36) to show the effect of compatibilizer type and amount on the composite properties in a more convenient way for polypropylene reinforced with small and large palm fibers. As seen in Figures (4.33) and (4.34) composite modulus increases with fiber content for composites with any of the two compatibilizers (Epolene E-43 and G-3003) added at various amounts (2, 4 and 6 wt%) as well as for the ones with no compatibilizer. As also discussed above this is an indication for the existence of adhesion between palm fibers and polypropylene matrix even in the composites without any compatibilizer.

However the use of compatibilizer might have increased the adhesion strength which is not that clear in the plots of modulus vs fiber content (considering also the scatter in data) but this fact is quite clear in Figures (4.35) and (4.36) which present plots of tensile strength vs fiber content for uncompatibilized and compatibilized palm-polypropylene composite with small and large fibers. As seen and also discussed previously there is a significant improvement in tensile strength with addition of any of the two compatibilizers while the greatest improvement is resulted with compatibilizer Epolene G-3003. In general as the compatibilizer content increases the composite strength also increases. Addition of 6 wt% compatibilizer Epolene G-3003 into the composite having 40 wt% palm fiber results in a composite strength (about 32 MPa for small fibers and 30 MPa for large fibers) close to that of polypropylene (about 34 MPa).

Figures (4.37)-(4.38) show the effect of compatibilizer type and amount on the composite elongation at break. It is seen that the addition of Epolene E-43 and G 3003 does not significantly affect our composite elongation.

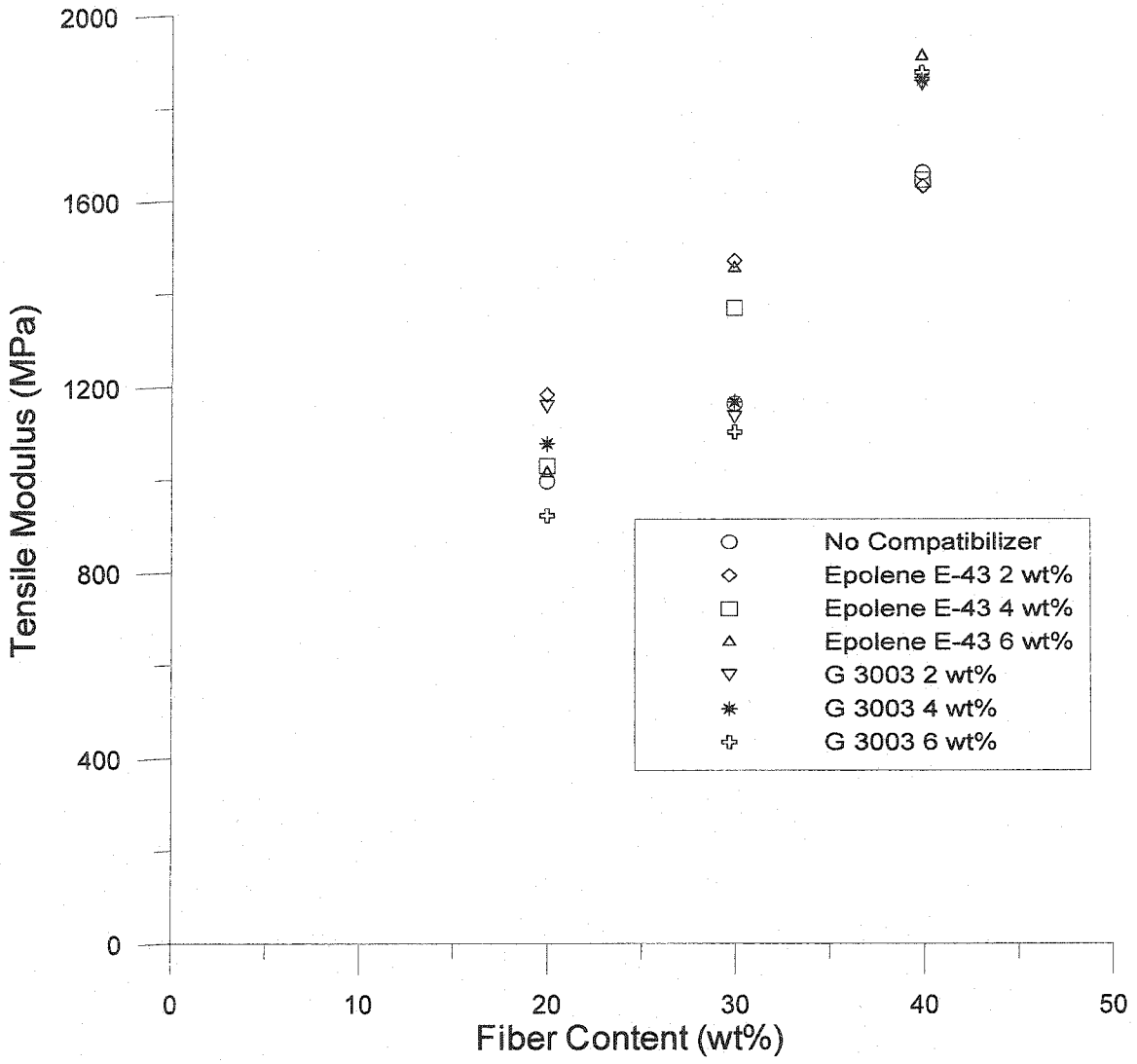


Figure (4.33) Tensile modulus vs fiber content for polypropylene reinforced with large palm fibers without any treatment and with utilizing compatibilizer Epolene E-43 or G-3003 at various amounts.

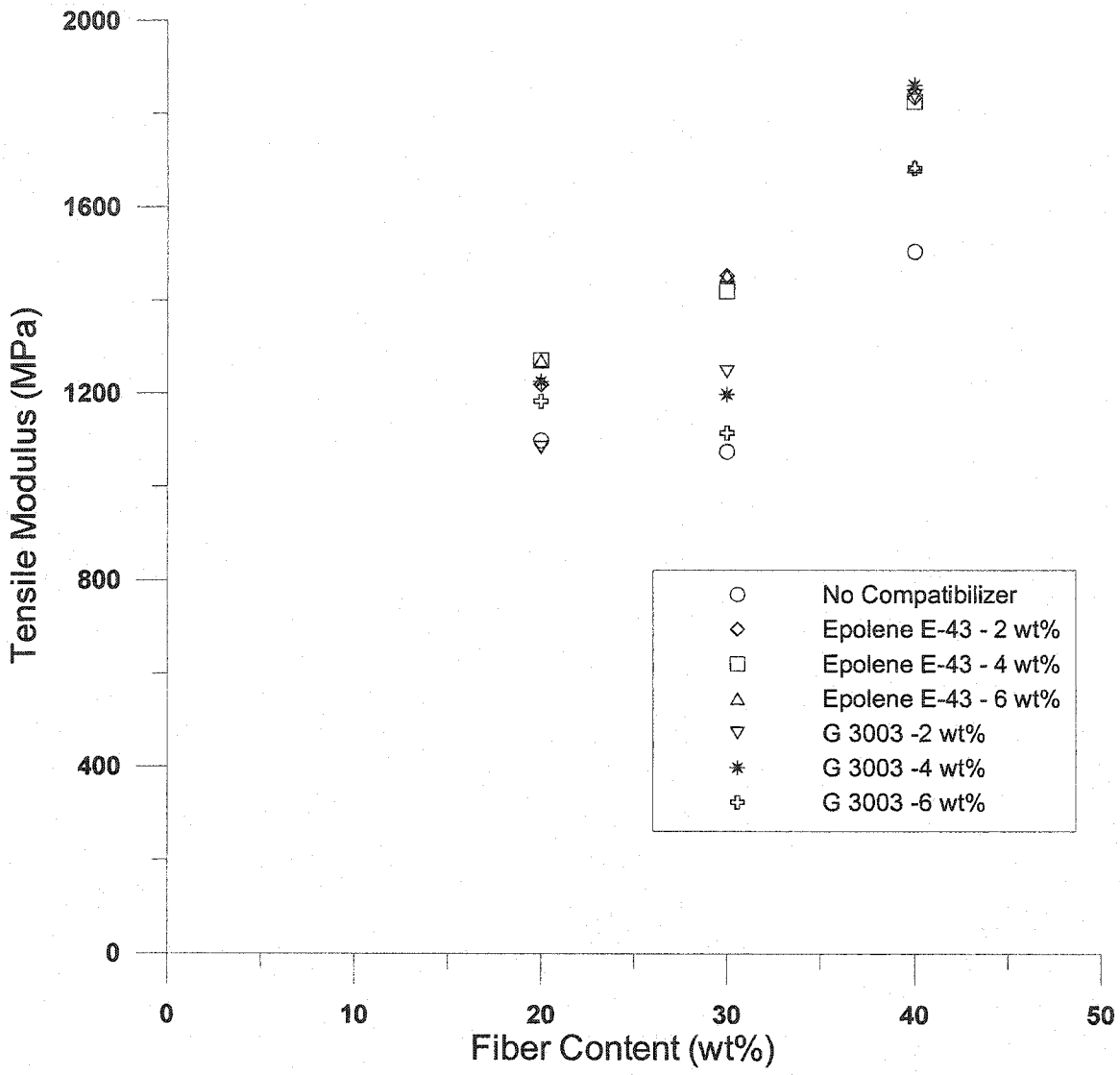


Figure (4.34) Tensile modulus vs fiber content for polypropylene reinforced with small palm fibers without any treatment and with utilizing compatibilizer Epolene E-43 or G-3003 at various amounts.

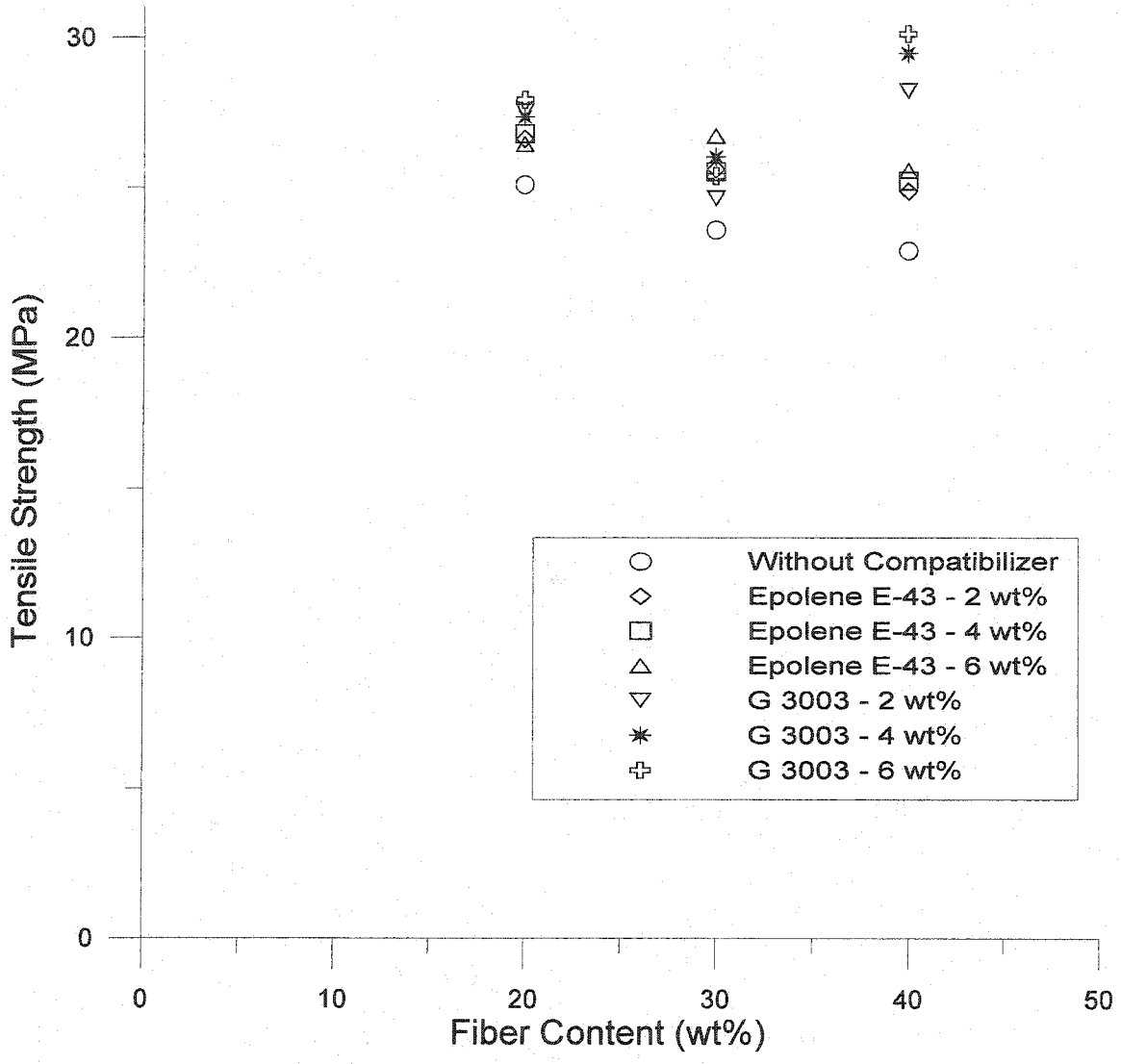


Figure (4.35) Tensile strength vs fiber content for polypropylene reinforced with large palm fibers without any treatment and with utilizing compatibilizer Epolene E-43 or G 3003 at various amounts.

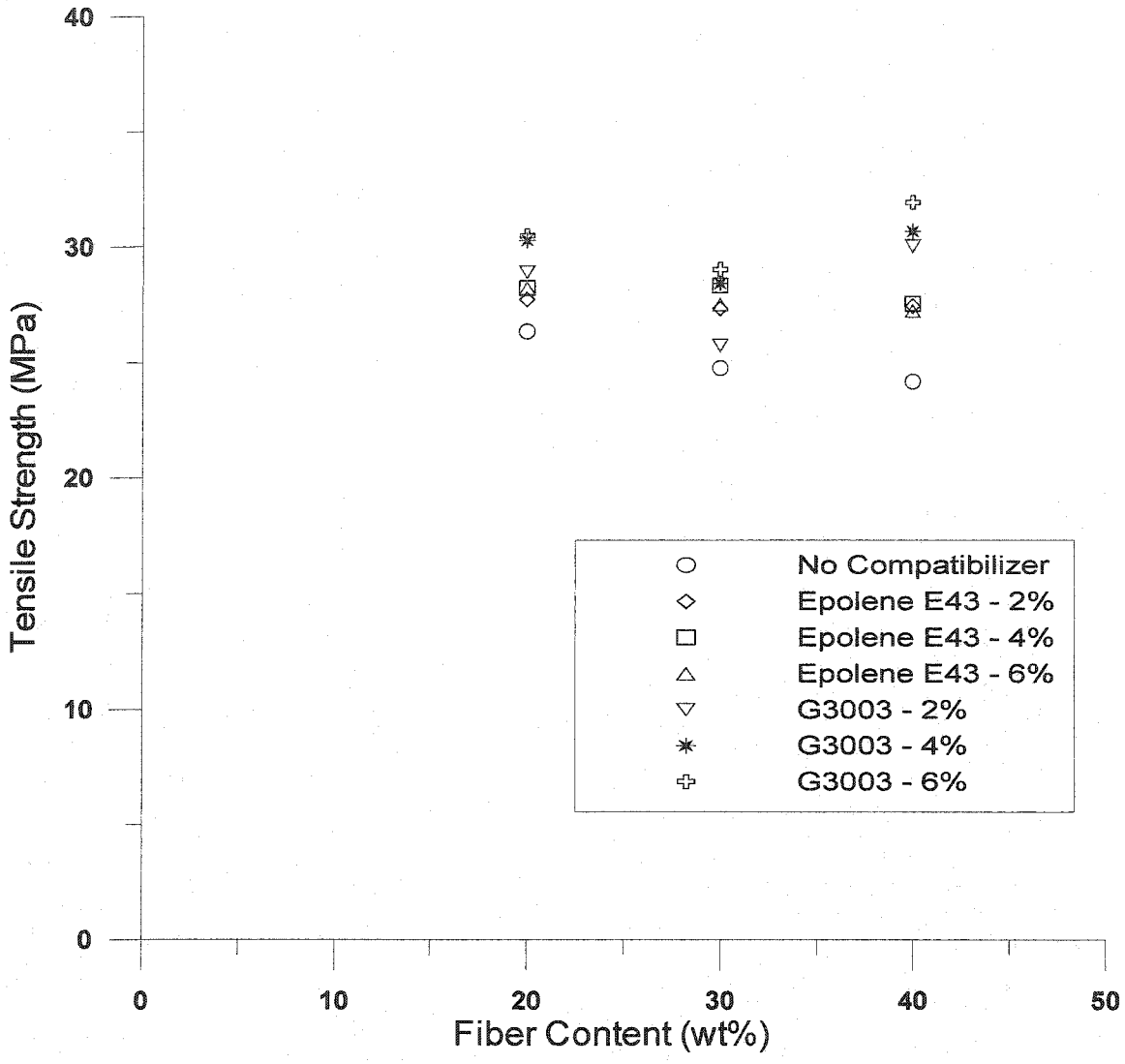


Figure (4.36) Tensile strength vs fiber content for polypropylene reinforced with small palm fibers without any treatment and with utilizing compatibilizer Epolene E-43 or G-3003 at various amounts.

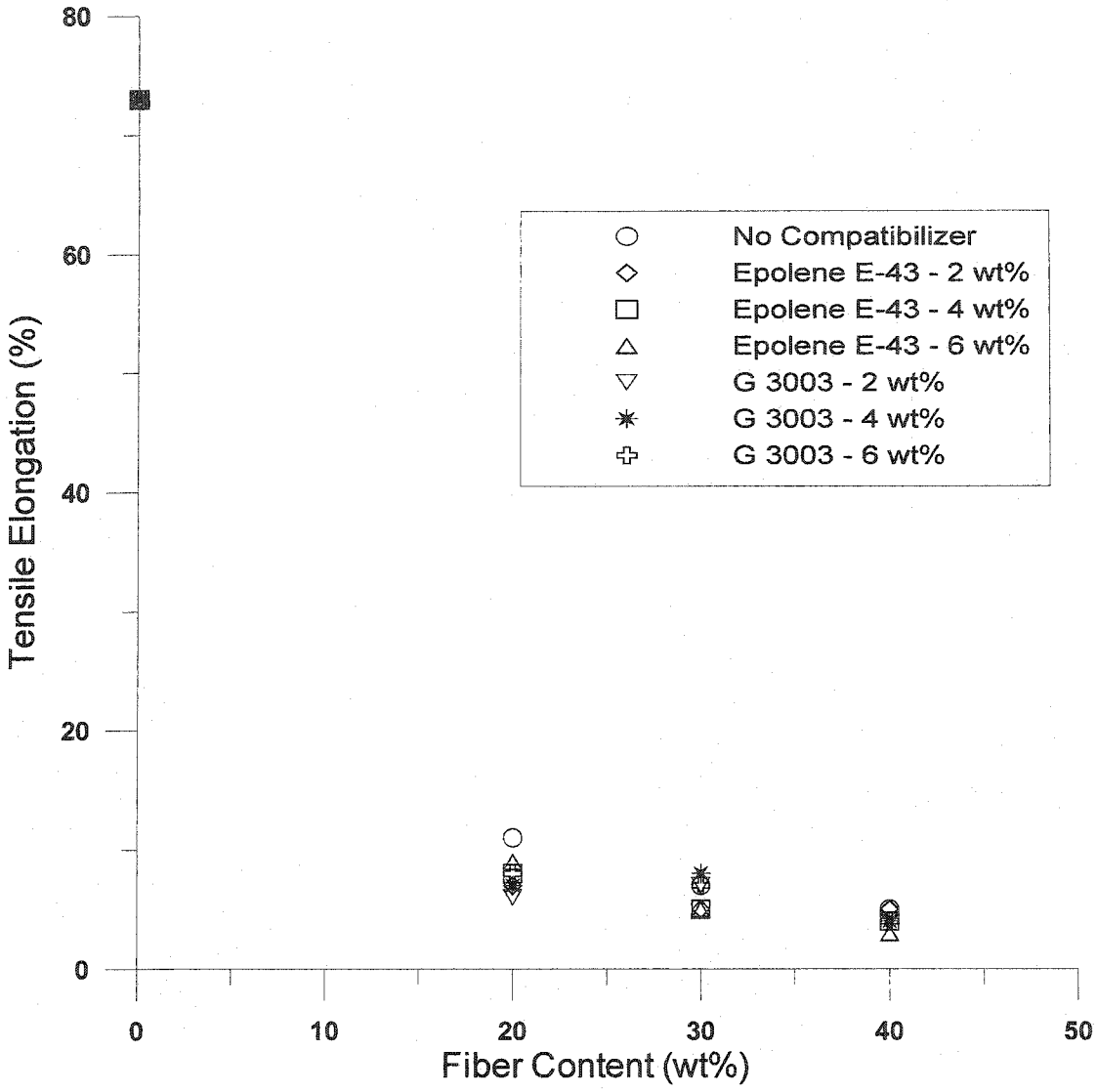


Figure (4.37) Tensile elongation vs fiber content for polypropylene reinforced with large palm fibers without any treatment and with utilizing compatibilizer Epolene E-43 or G-3003 at various amounts.

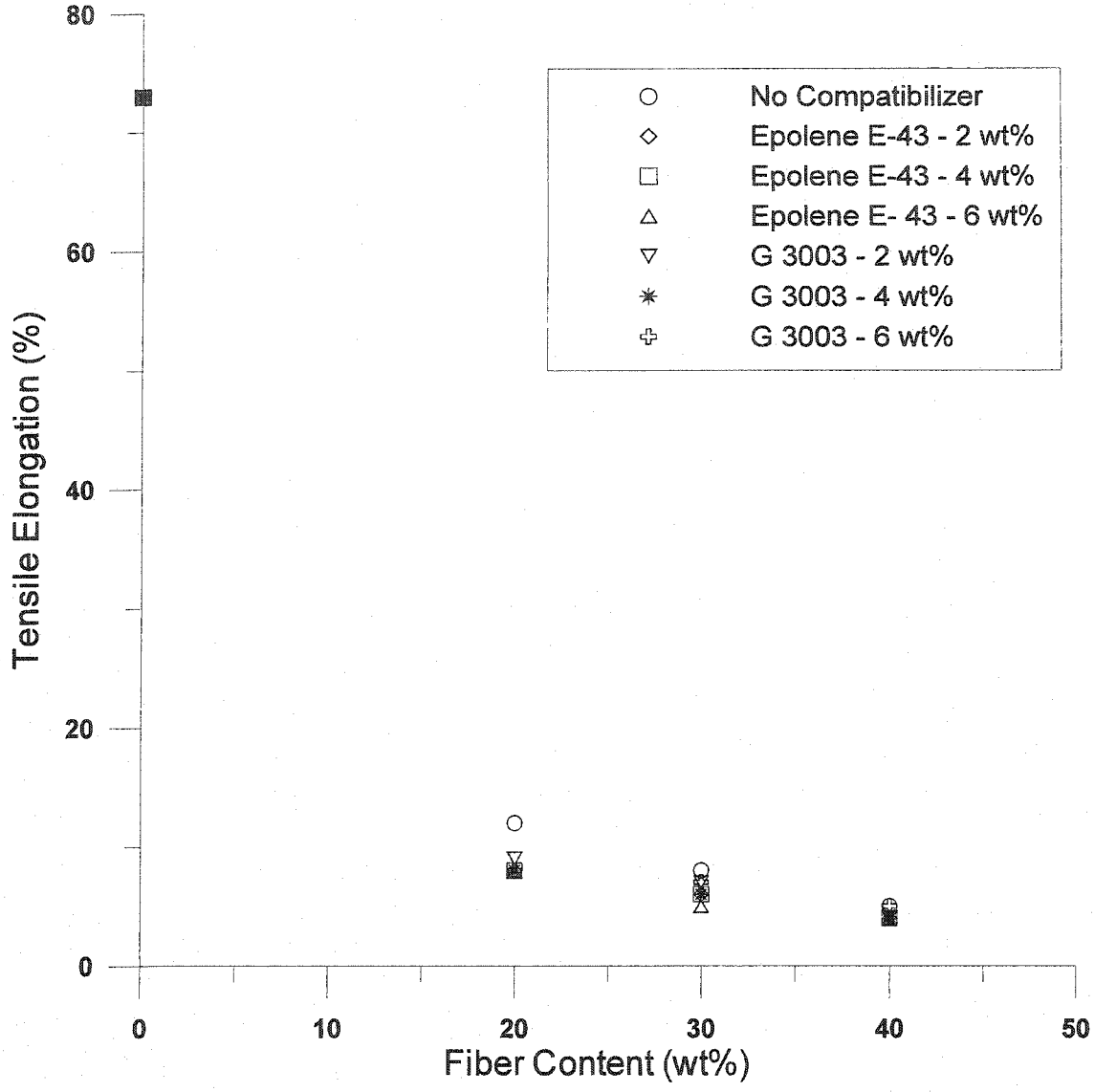


Figure (4.38) Tensile elongation vs fiber content for polypropylene reinforced with small palm fibers without any treatment and with utilizing compatibilizer Epolene E-43 or G-3003 at various amounts

The data presented above can be rearranged to be able to discuss the effect of varying compatibilizer on the mechanical properties of the polypropylene composite. Figure (4.39) shows the tensile strength versus compatibilizer content for Epolene E-43 with varying fiber loading. It is seen as a general trend for the two fiber length distributions that increasing amount of compatibilizer content increases the tensile strength which is indication of a probable increase in fiber-matrix adhesion strength.

Figure (4.40) show the tensile strength against compatibilizer content for Epolene G-3003 with varying fiber length distribution (both small and large). In general a similar trend of increasing tensile strength with increasing the compatibilizer content is observed.

Figure (4.41) shows the tensile modulus versus compatibilizer content of Epolene E-43. There is no significant effect of compatibilizer content on composite modulus. This is understandable since improvement in fiber-matrix adhesion strength with compatibilizer addition is not expected to improve composite modulus as explained previously (existence of adhesion between fiber and matrix would result in the same modulus whether it is weak or strong).

Figure (4.42) present the tensile modulus versus compatibilizer content with varying fiber loading for G-3003 as compatibilizer. The result was not any different.

Figure (4.43) represents the tensile elongation against the compatibilizer content of Epolene E 43. The presence of compatibilizer does not affect the composite elongation

significantly. A similar trend is observed in Figure (4.44) for Epolene G 3003. There is no improvement nor there is drastic decrease with the addition of compatibilizer.

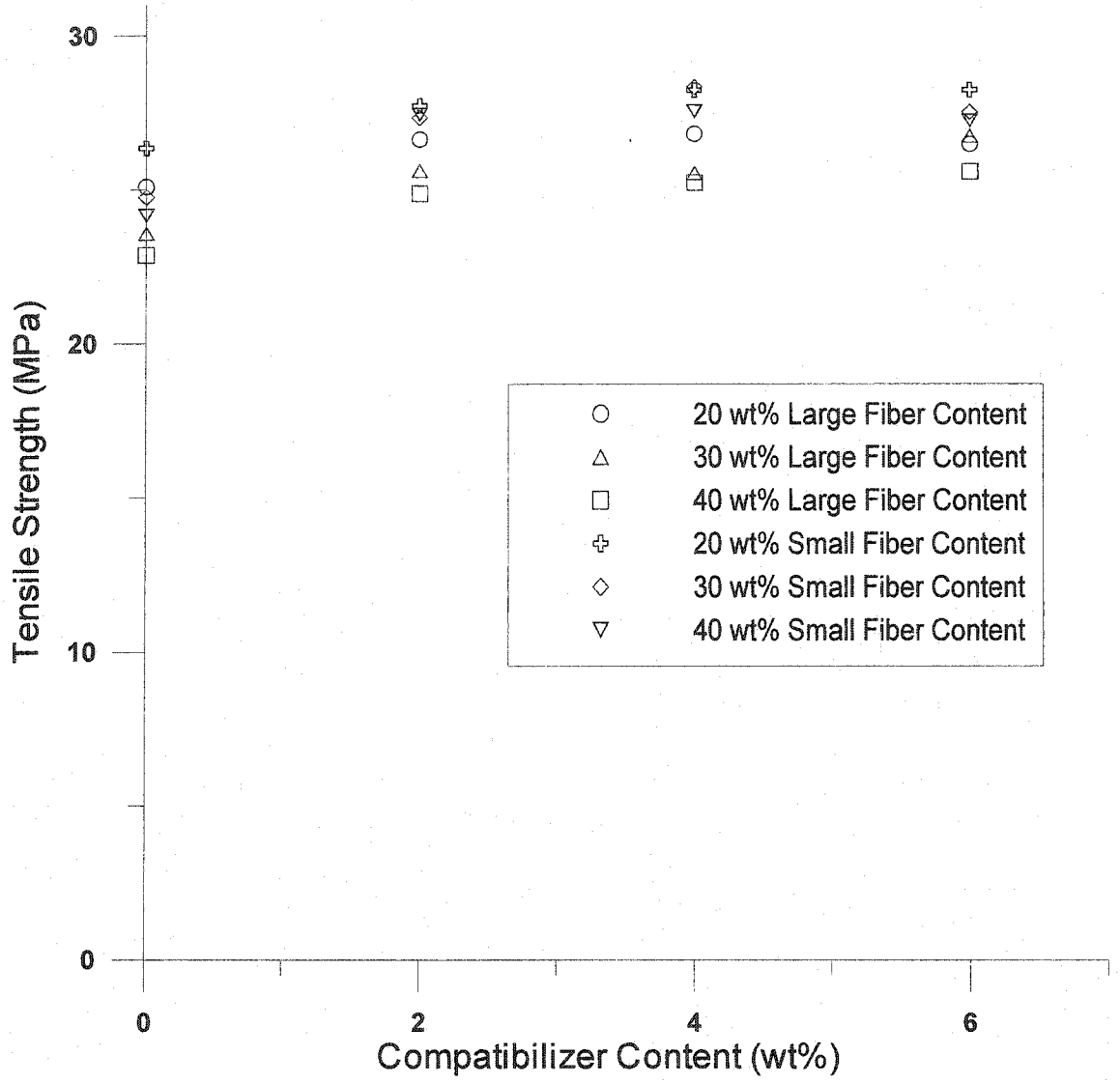


Figure (4.39) Tensile strength vs compatibilizer content for polypropylene reinforced with palm fibers without any treatment and with utilizing compatibilizer Epolene E-43 at various amounts.

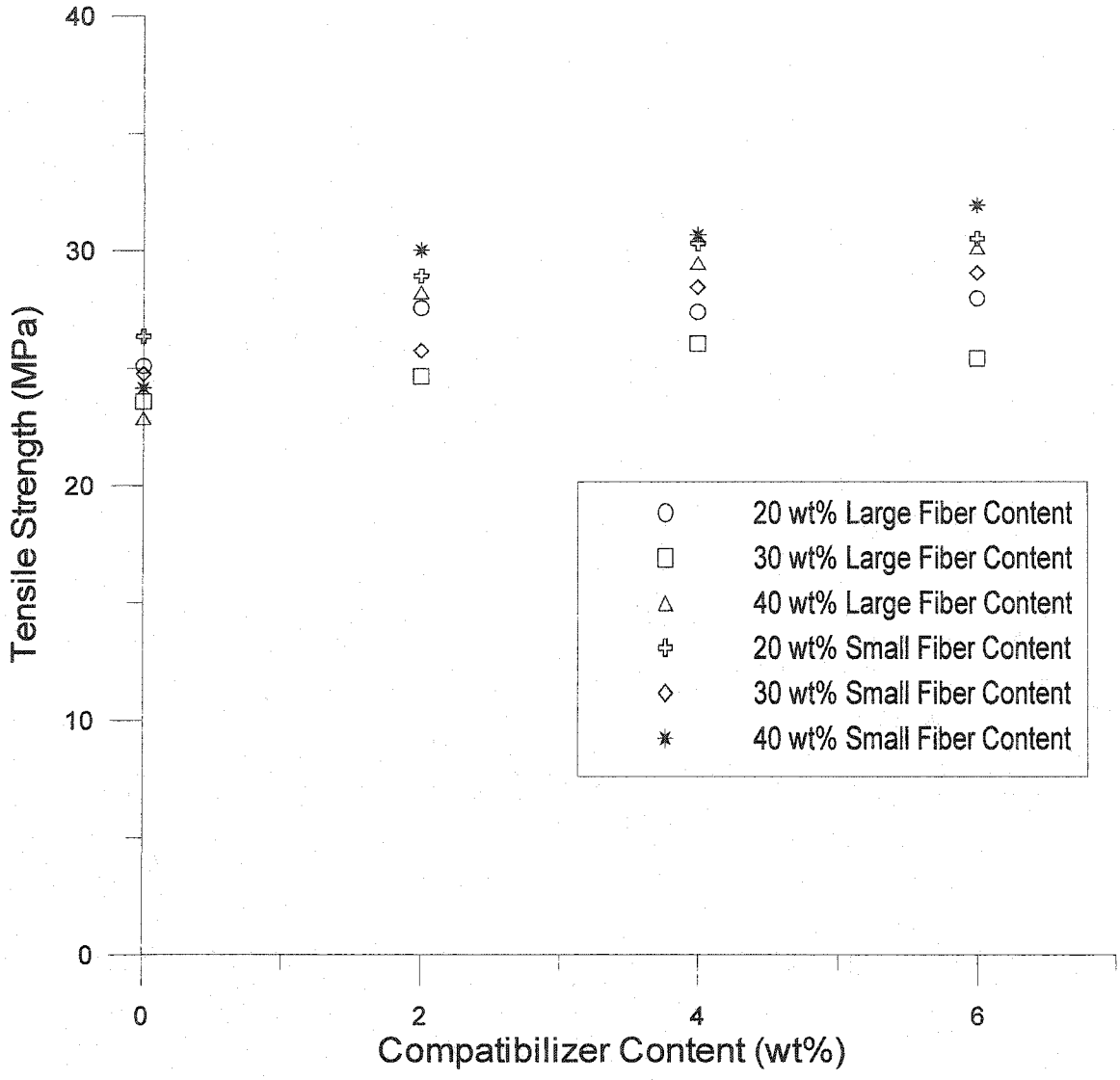


Figure (4.40) Tensile strength vs compatibilizer content for polypropylene reinforced with palm fibers without any treatment and with utilizing compatibilizer G-3003 at various amounts.

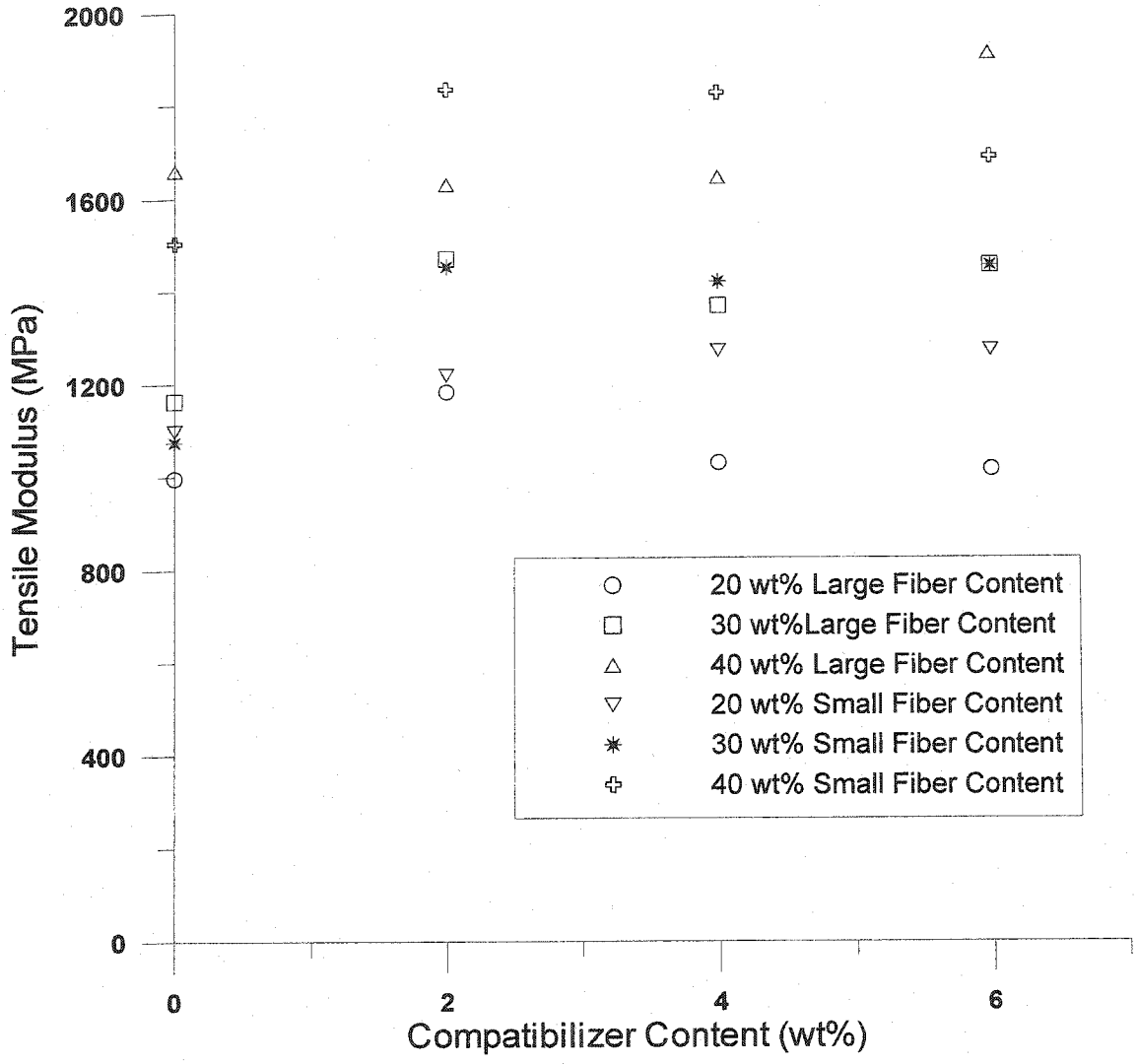


Figure (4.41) Tensile modulus vs compatibilizer content for polypropylene reinforced with palm fibers without any treatment and with utilizing compatibilizer Epolene E-43 at various amounts.

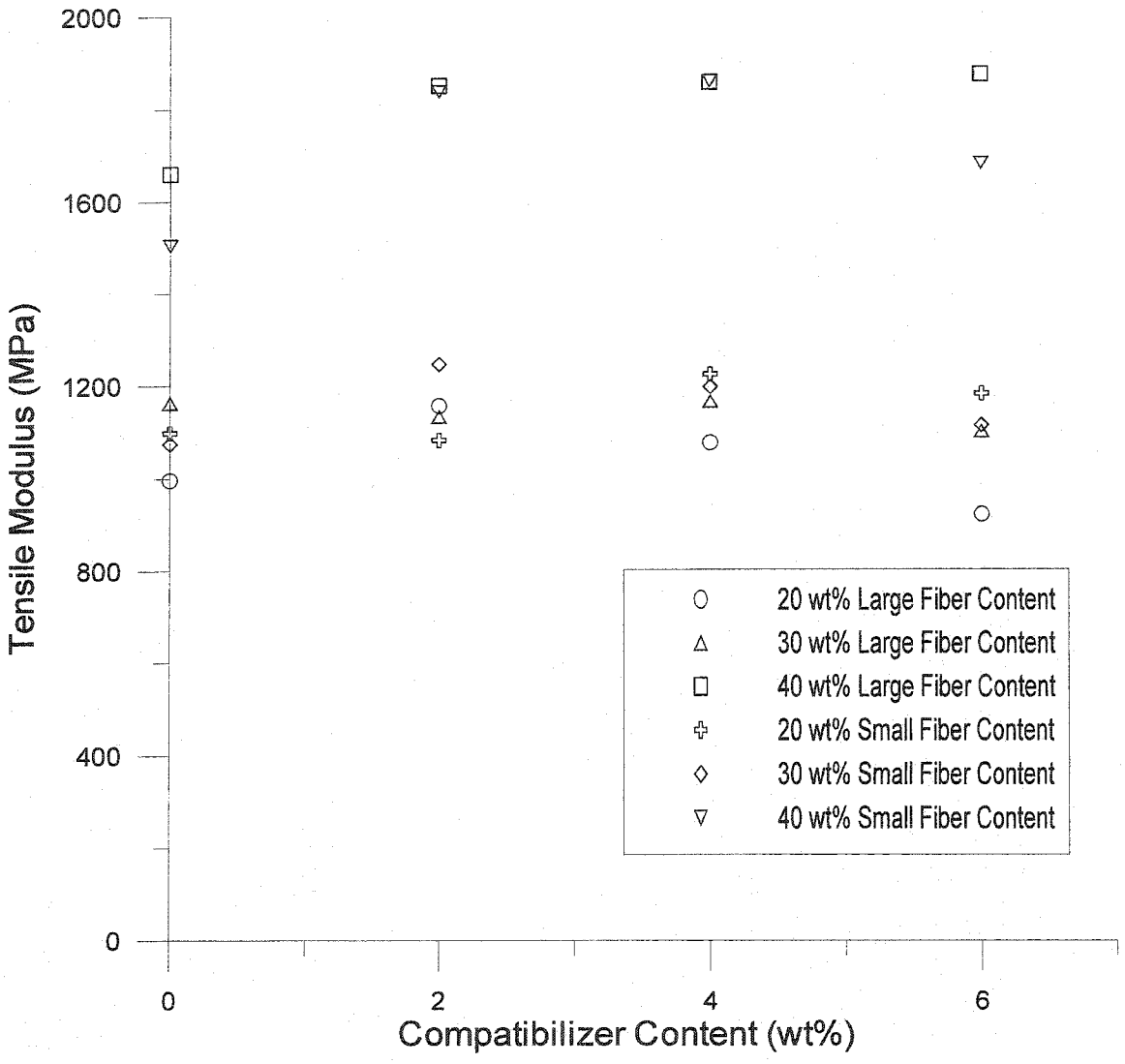


Figure (4.42) Tensile modulus vs compatibilizer content for polypropylene reinforced with palm fibers without any treatment and with utilizing compatibilizer G 3003 at various amounts.

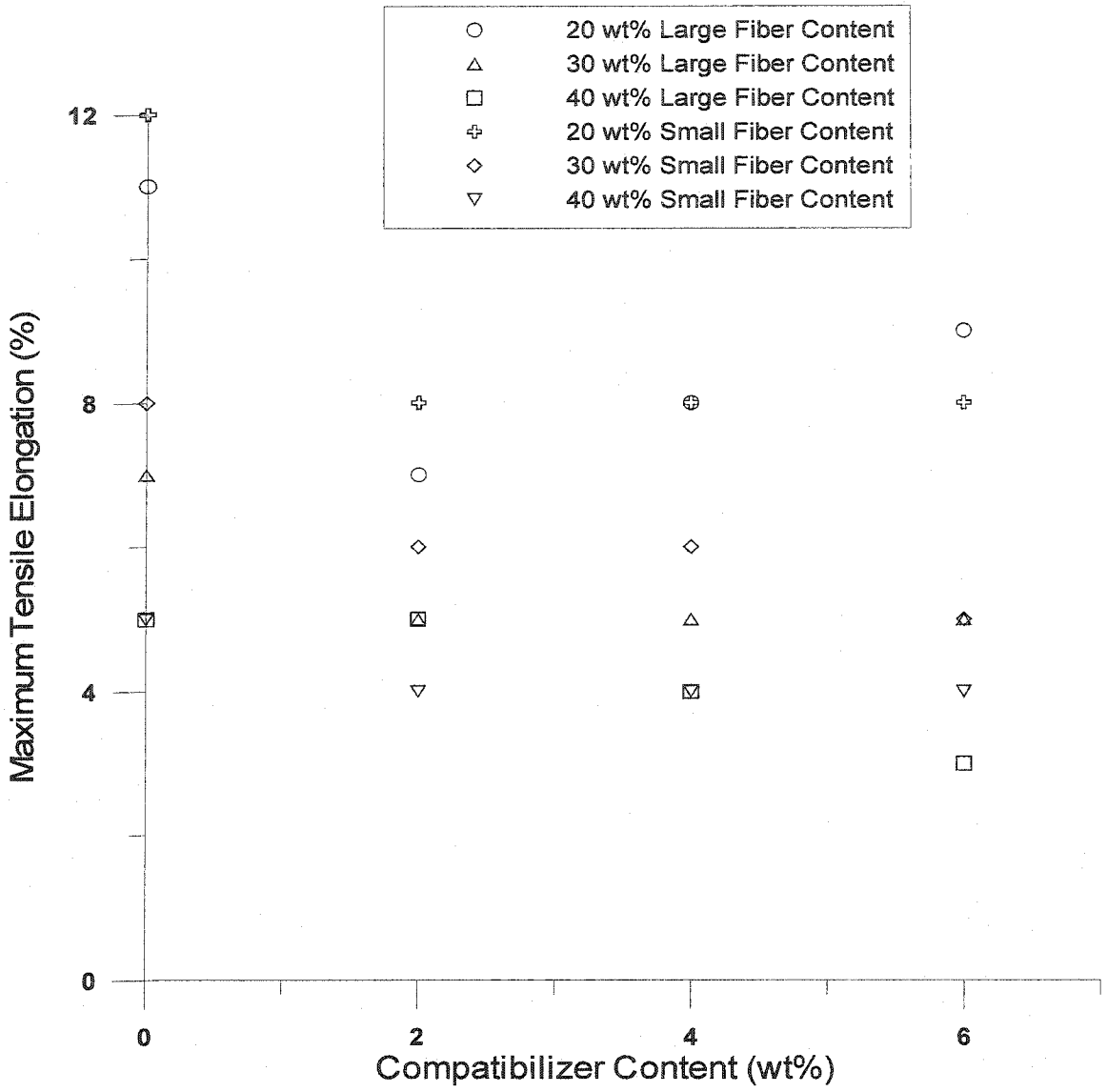


Figure (4.43) Tensile elongation vs compatibilizer content for polypropylene reinforced with palm fibers without any treatment and with utilizing compatibilizer Epolene E-43 at various amounts.

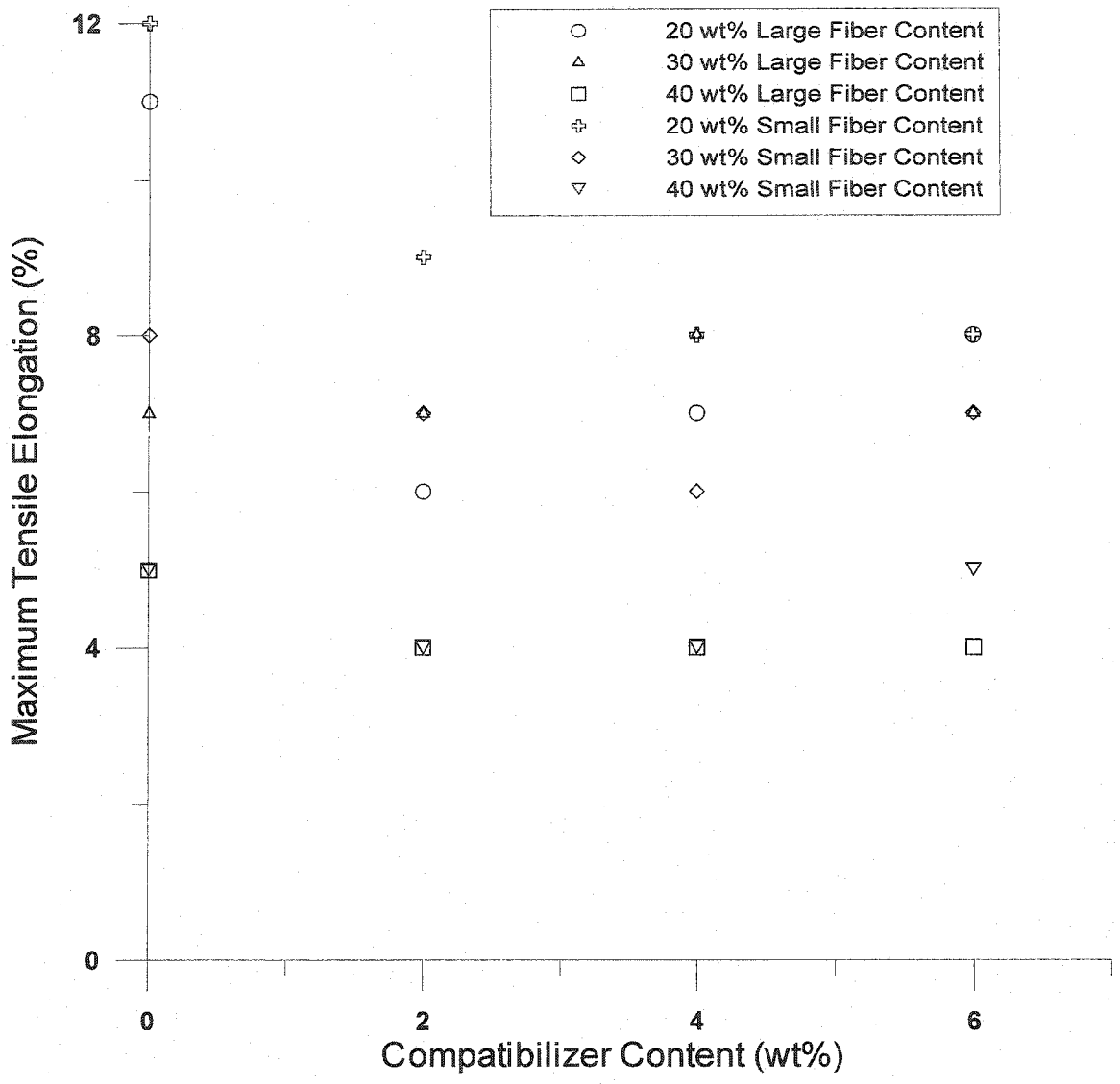


Figure (4.44) Tensile elongation vs compatibilizer content for polypropylene reinforced with palm fibers without any treatment and with utilizing compatibilizer G 3003 at various amounts.

4.3 Morphological Characterization

Fracture surfaces of the mechanically tested composite specimens were also analyzed by scanning electron microscopy (SEM).

It was seen that polypropylene composites containing no compatibilizer resulted in rough fracture surfaces with extensive fiber pullout [Figures (4.45) and (4.46)]. This is an indication of poor bonding between the fiber and the polypropylene matrix (as compared to that of the composites with compatibilizer treated fibers, discussion of which will follow). This explains why composites with untreated fibers perform worse than those with compatibilizer treated fibers.

Once we incorporated the compatibilizer Epolene E-43 we see that instead of fiber pullout, a smoother fracture surface (still with some roughness) is observed [Figures (4.47) and 4.48)] as compared to that of the composite without the compatibilizer [Figures (4.45) and (4.48)]. The reason for this observation is probably that the incorporation of the compatibilizer E-43 has enhanced the interfacial adhesion between the fibers and matrix. That is why a higher load was transferred onto the fibers, hence a higher composite strength was observed.

Composites with compatibilizer Epolene G-3003 produced even smoother fracture surfaces with no significant fiber pullout [Figures (4.49) and (4.50)] which is an indication for improved adhesion between the palm fibers and the polypropylene matrix.

This is consistent with the results of the mechanical tests performed on the uncompatibilized and compatibilized composites. Composites with compatibilizer G 3003 were the strongest.

Figure (4.45) SEM micrograph of a fractured surface of a 30 wt% fiber palm-polypropylene composite with no compatibilizer.

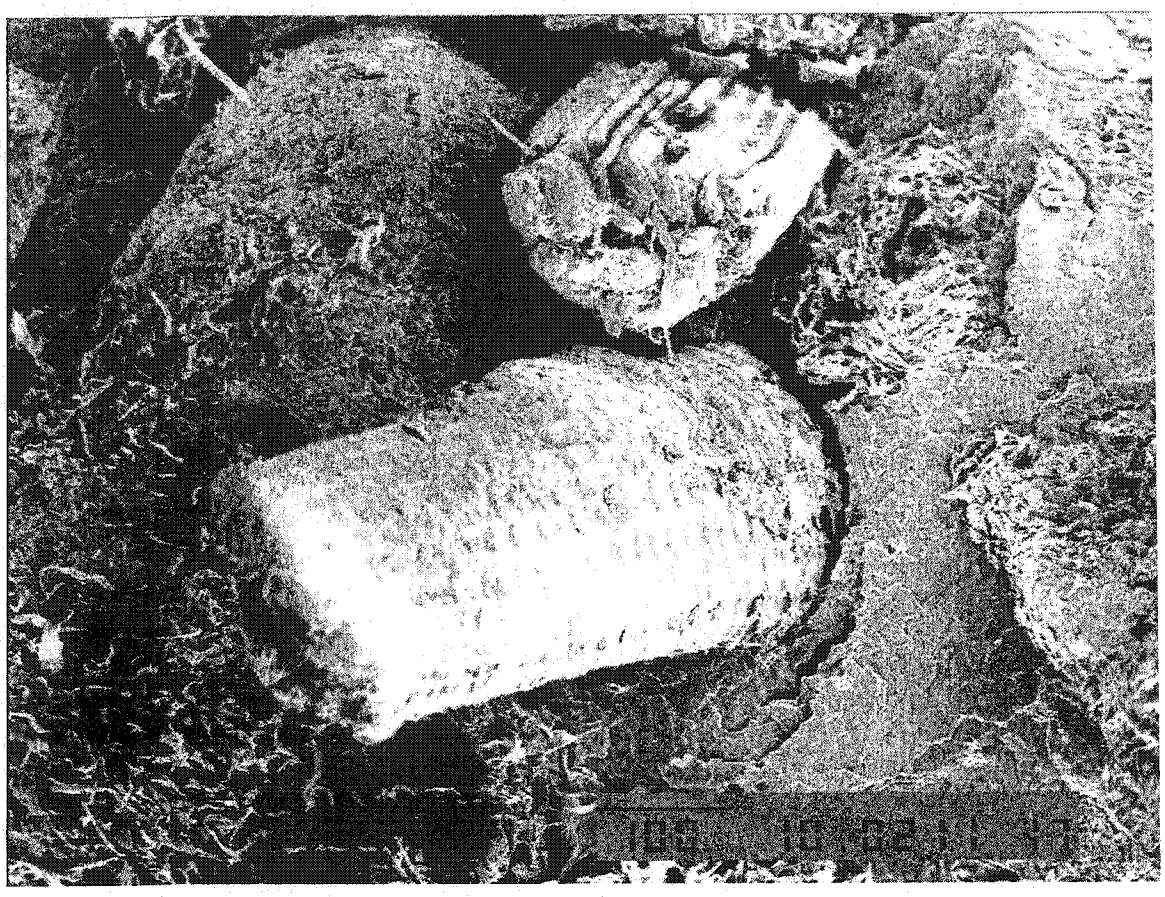


Figure (4.46) SEM micrograph of a fractured surface of a 30 wt% fiber palm-polypropylene composite with no compatibilizer.

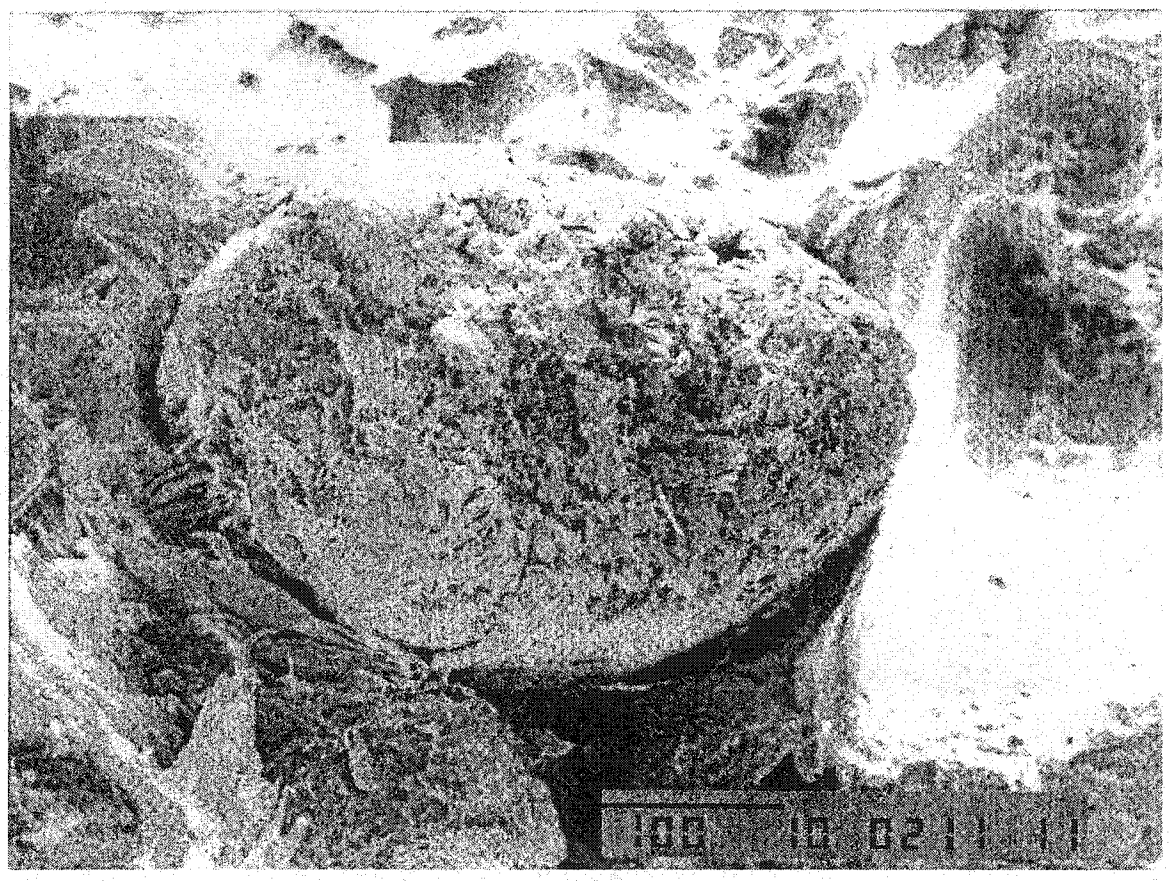


Figure (4.47) SEM micrograph of a fractured surface of a 30 wt% fiber palm-polypropylene composite compatibilized with 6 wt% Epolene E-43.

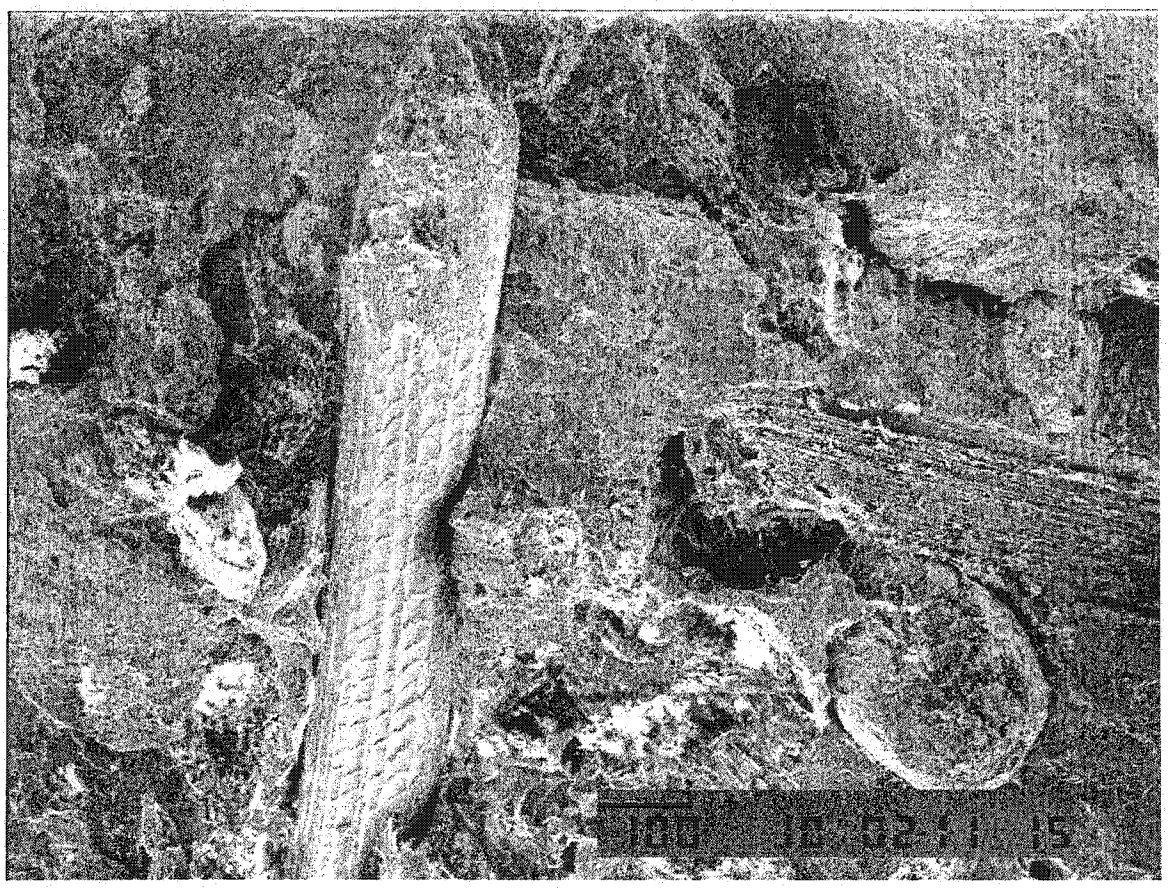


Figure (4.48) SEM micrograph of a fractured surface of a 30 wt% fiber palm-polypropylene composite compatibilized with 6wt% Epolene E-43.

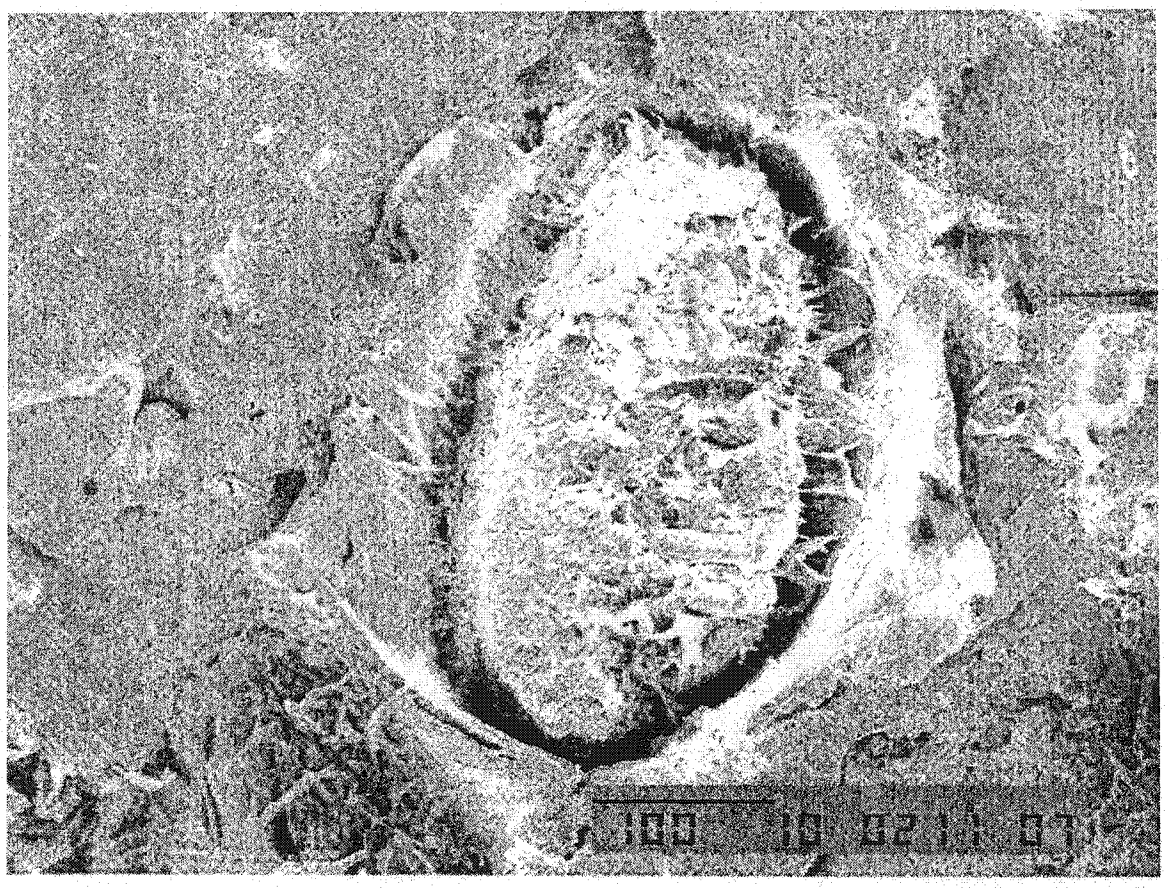


Figure (4.49) SEM micrograph of a fractured surface of a 30 wt% fiber palm-polypropylene composite compatibilized with 6 wt% Epolene G-3003.

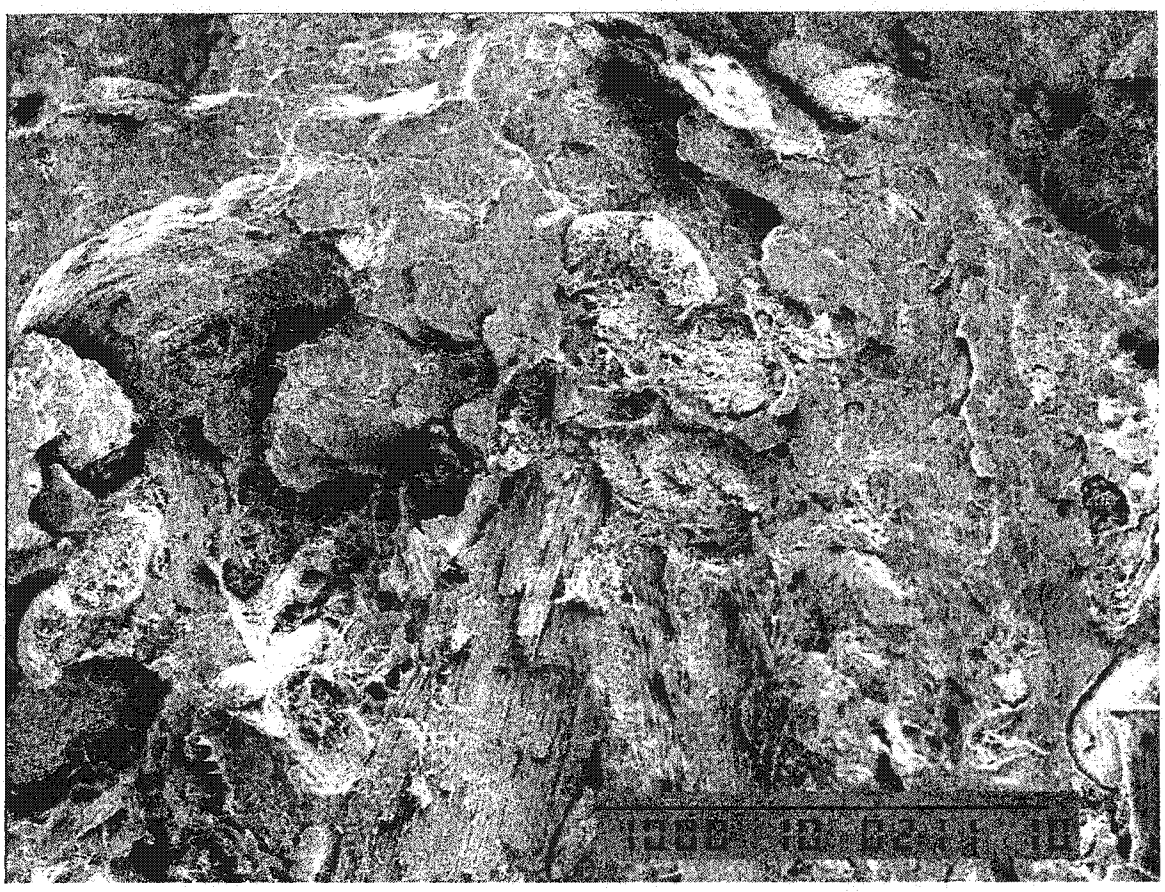


Figure (4.50) SEM micrograph of a fractured surface of a 30 wt% fiber palm-polypropylene composite with Epolene 6 wt% G-3003.

4.4 DSC Study

The samples were prepared in order to conform with the requirements of the equipment (Mettler Toledo DSC 822C Star Thermal Analysis System). The samples weight was approximately 5 mg. Once the samples were prepared, they were subjected to same sample history by first cooling the samples from 220°C to 20°C by decreasing the temperature at the rate of 10°C per minute. Thereafter the samples were heated at 10°C per minute from 20°C to 220°C.

The thermograms of the samples tested are given in the Appendix (Section B). In order to investigate the influence of fiber content on crystallinity, DSC was used to measure the percent crystallinity (X_c) of polypropylene in the PP/fiber composites as a function of fiber content. As shown in Figure (4.51), increasing fiber content was found to increase X_c of the composite. This suggests that the crystalline cellulose fibers enhanced the chain folding of PP, possibly by providing a template for its crystallization. However, the increase in crystallinity was not significant enough to explain the increase in tensile strength and modulus of the compatibilized composite with fiber content.

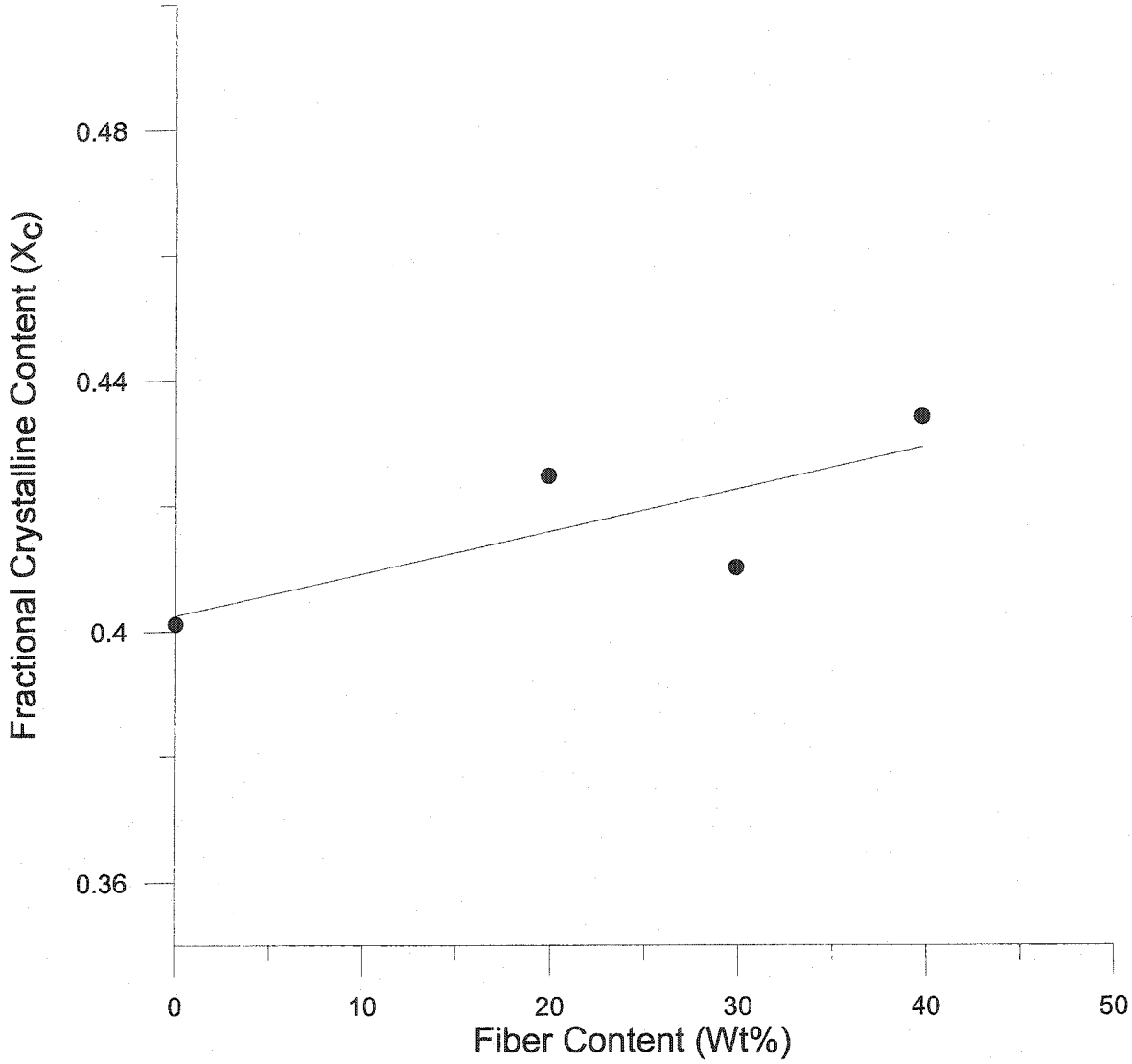


Figure (4.51) Effect of fiber content on fractional crystalline content of palm fiber / polypropylene composite.

4.5 FTIR Study

Fourier Transform Infrared Spectroscopy was performed to detect formation of any new functional group by the addition of the compatibilizer into the palm/polypropylene composite. From the Data sheet of Epolene G-3003, it is stated that this polypropylene is maleic anhydride grafted to minimize the phase separation between polar reinforcements/fillers and non-polar polymers in filled composites. Although the Data sheet of Epolene EP-43 only say that it is chemically modified polypropylene. But upon studying the transmission spectra of pure Epolene EP-43 (Figure 4.52b), peaks were observed at 1845 cm^{-1} and 1777 cm^{-1}. And we already know that anhydride functionality is expected to show absorption at 1865 cm^{-1} and 1782 cm^{-1}. Which clearly shows that Epolene EP-43 is also maleic anhydride grafted polypropylene. Transmission spectra of pure Polypropylene, Fiber, pure Epolene EP 43, pure Epolene G-3003, Palm37% / Polypropylene57% composite with 6% Epolene EP-43, Palm37% / Polypropylene 57% composite with 6% Epolene G-3003 are shown in Figures (4.52)- (4.57).

Now once the fiber is blended with polypropylene and compatibilizer, formation of ester linkage is expected. IR absorption of the ester group appears at around 1740cm^{-1}. However identification of the ester functionality is difficult in our case as the IR spectra of fiber exhibited absorption at 1735 cm^{-1} (which may be attributed to the presence of some kind of carboxylic functional groups).

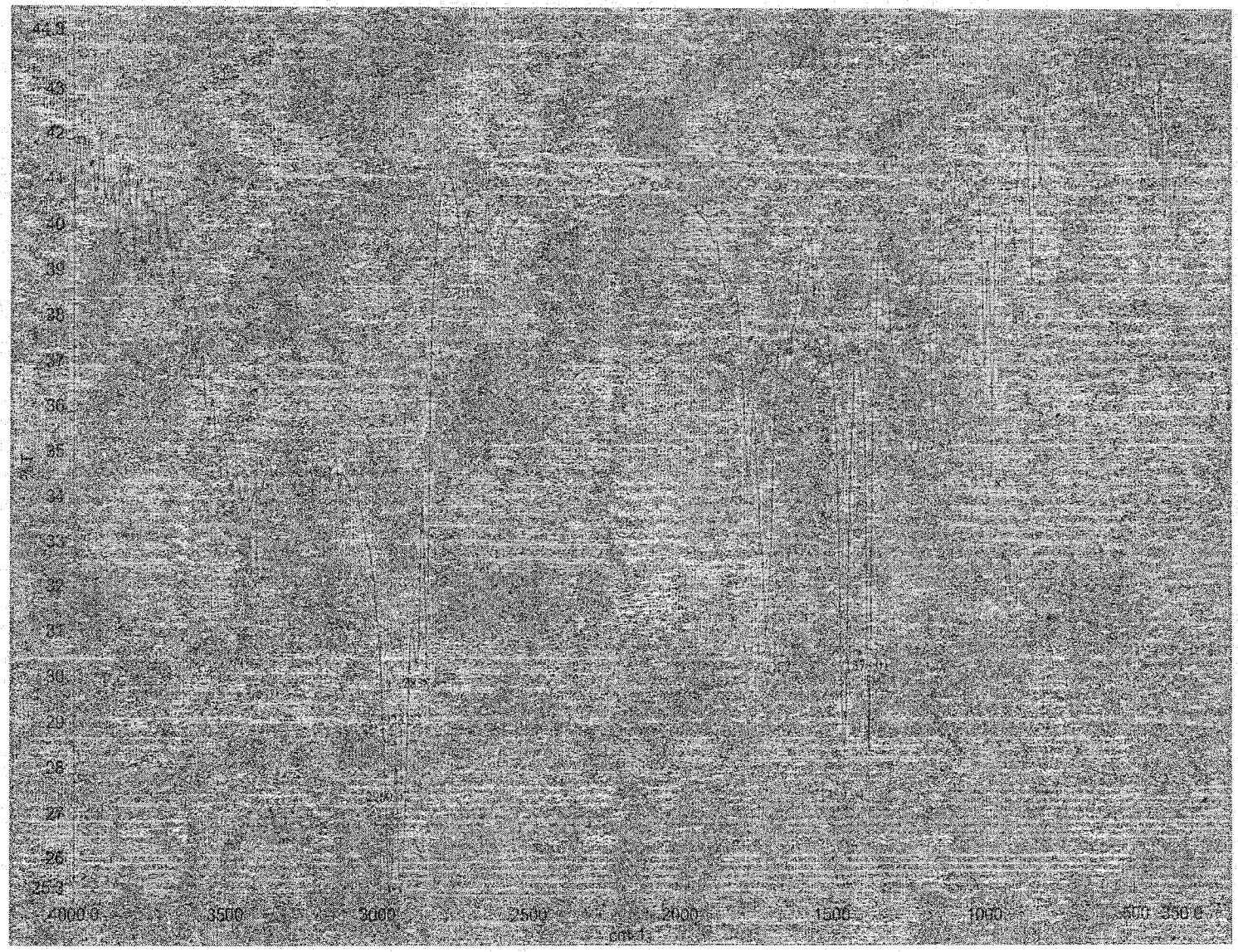


Figure (4.52a) Transmission spectra of pure Epolene EP-43.

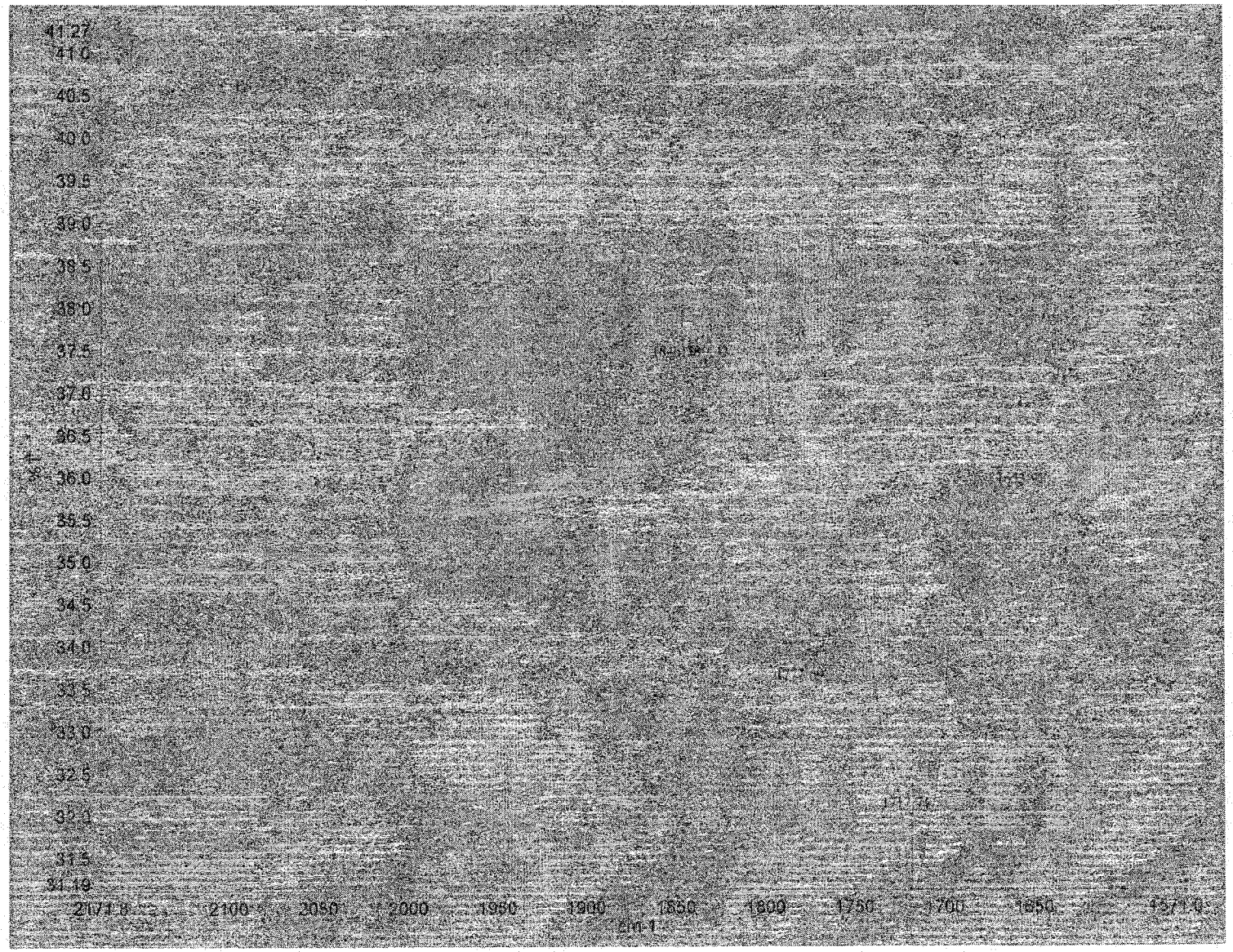


Figure (4.52b) Transmission spectra of the area of interest for Pure Epolene EP-43.

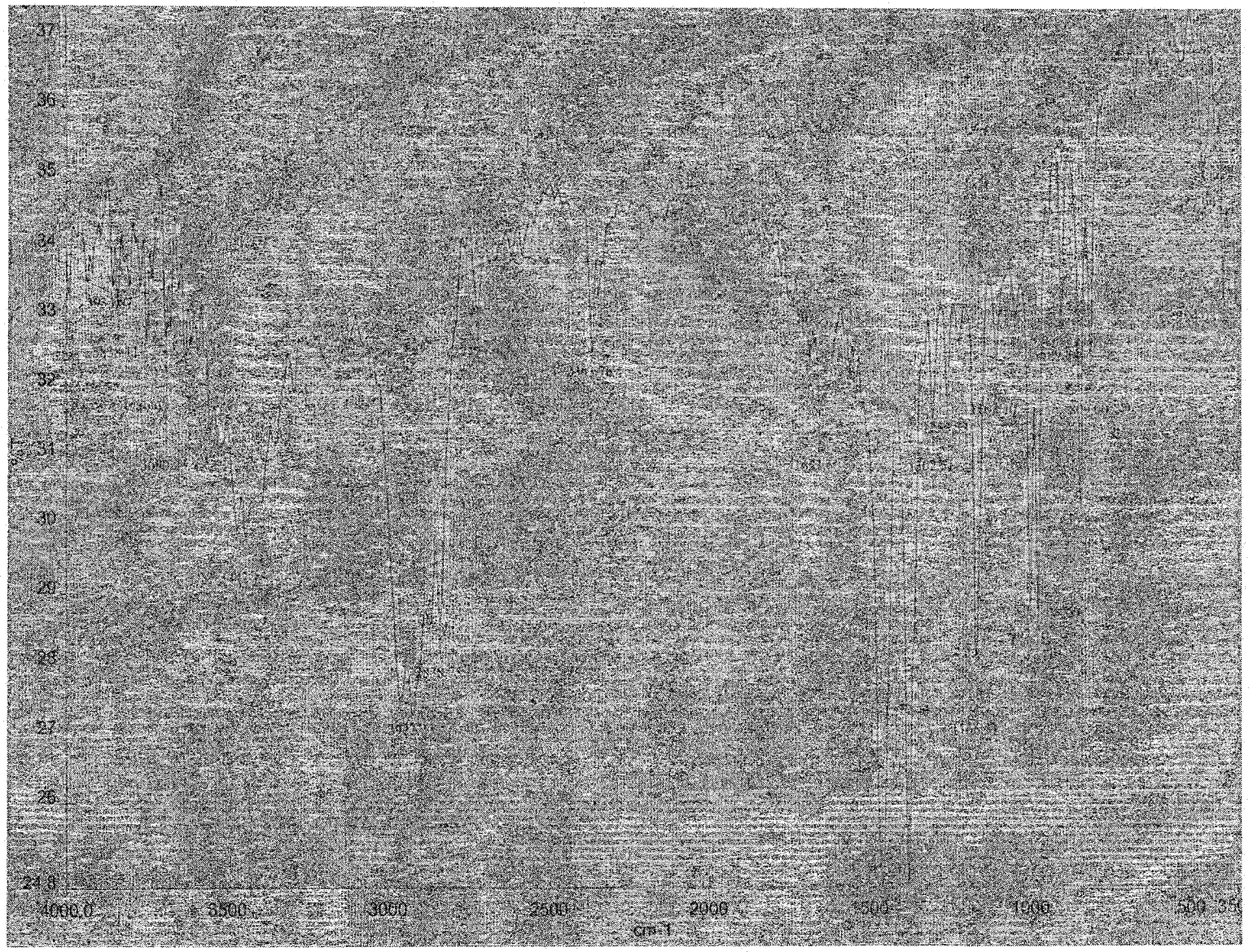


Figure (4.53) Transmission spectra of pure polypropylene.

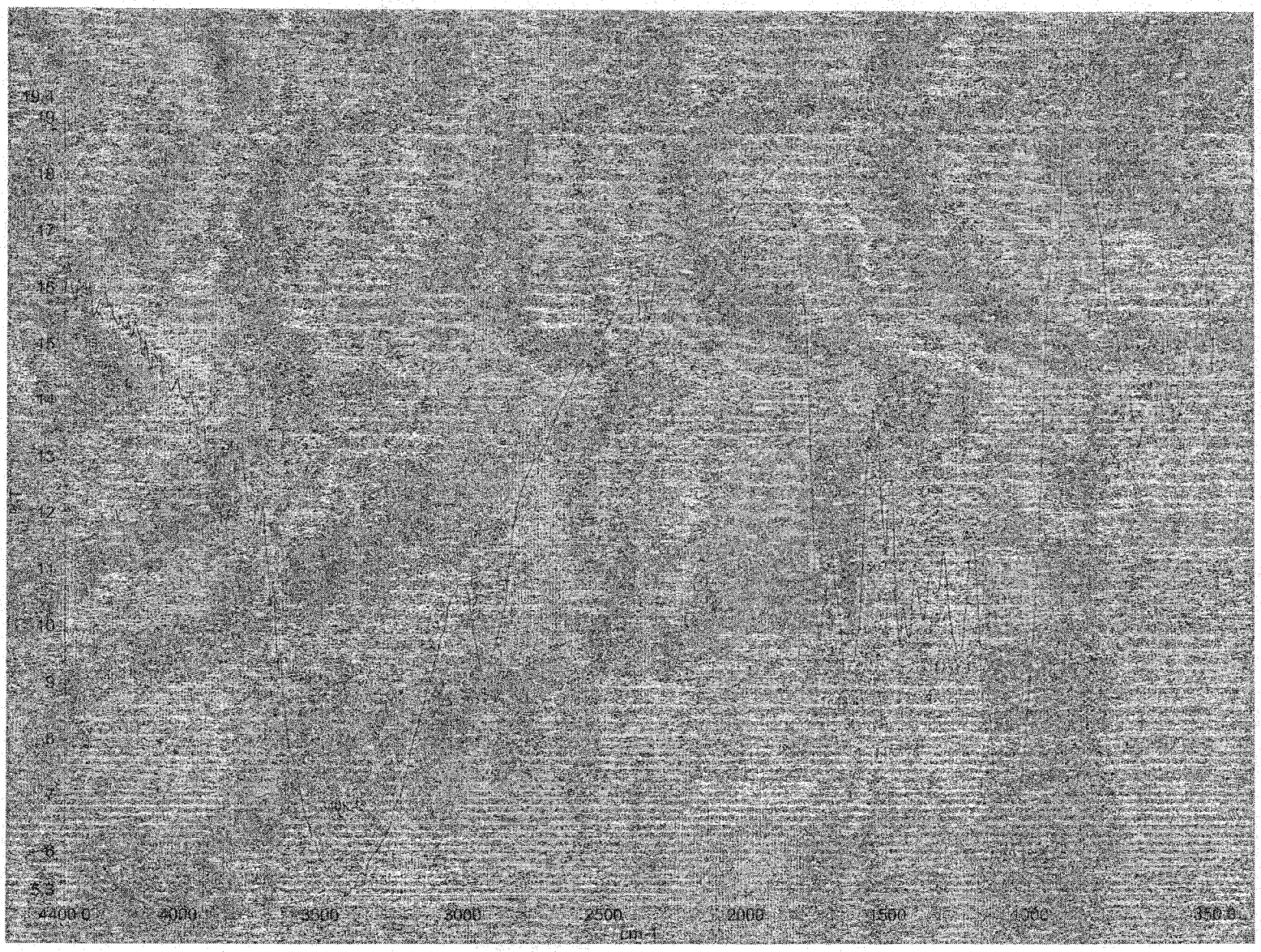


Figure (4.54) Transmission spectra of palm fiber.

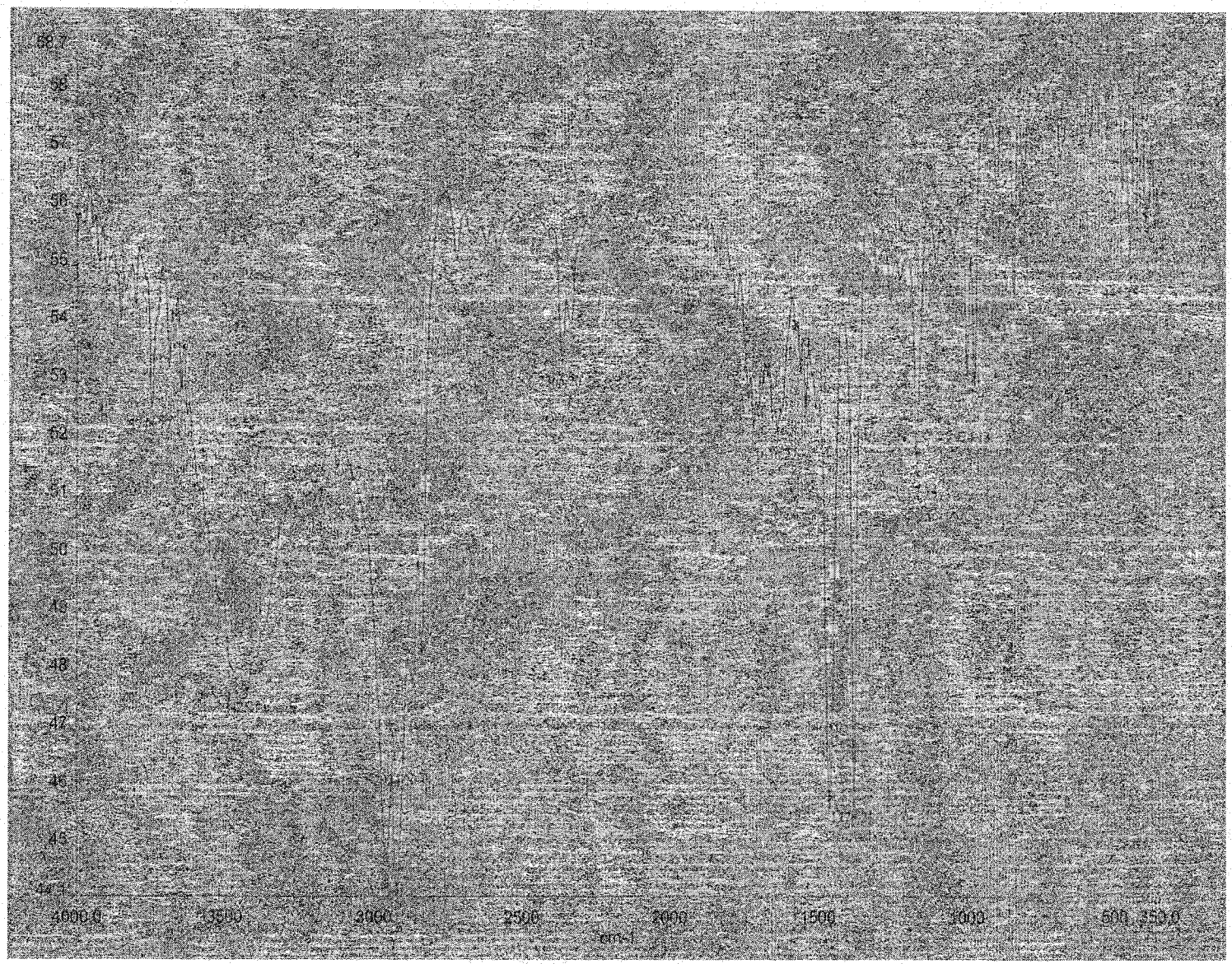


Figure (4.55a) Transmission spectra of Epolene G-3003.

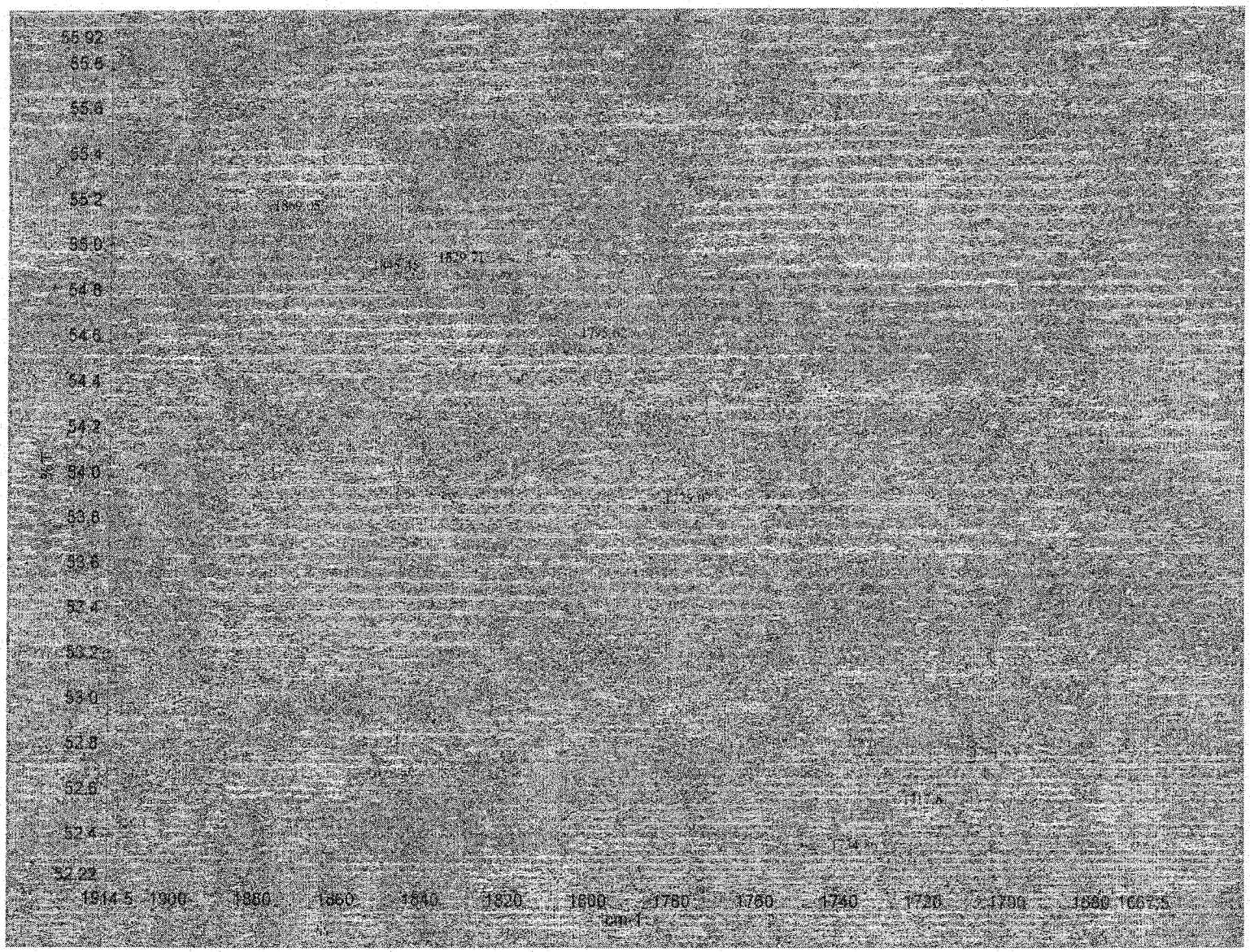


Figure (4.55b) Transmission spectra of the area of interest for pure Epolene G-3003.

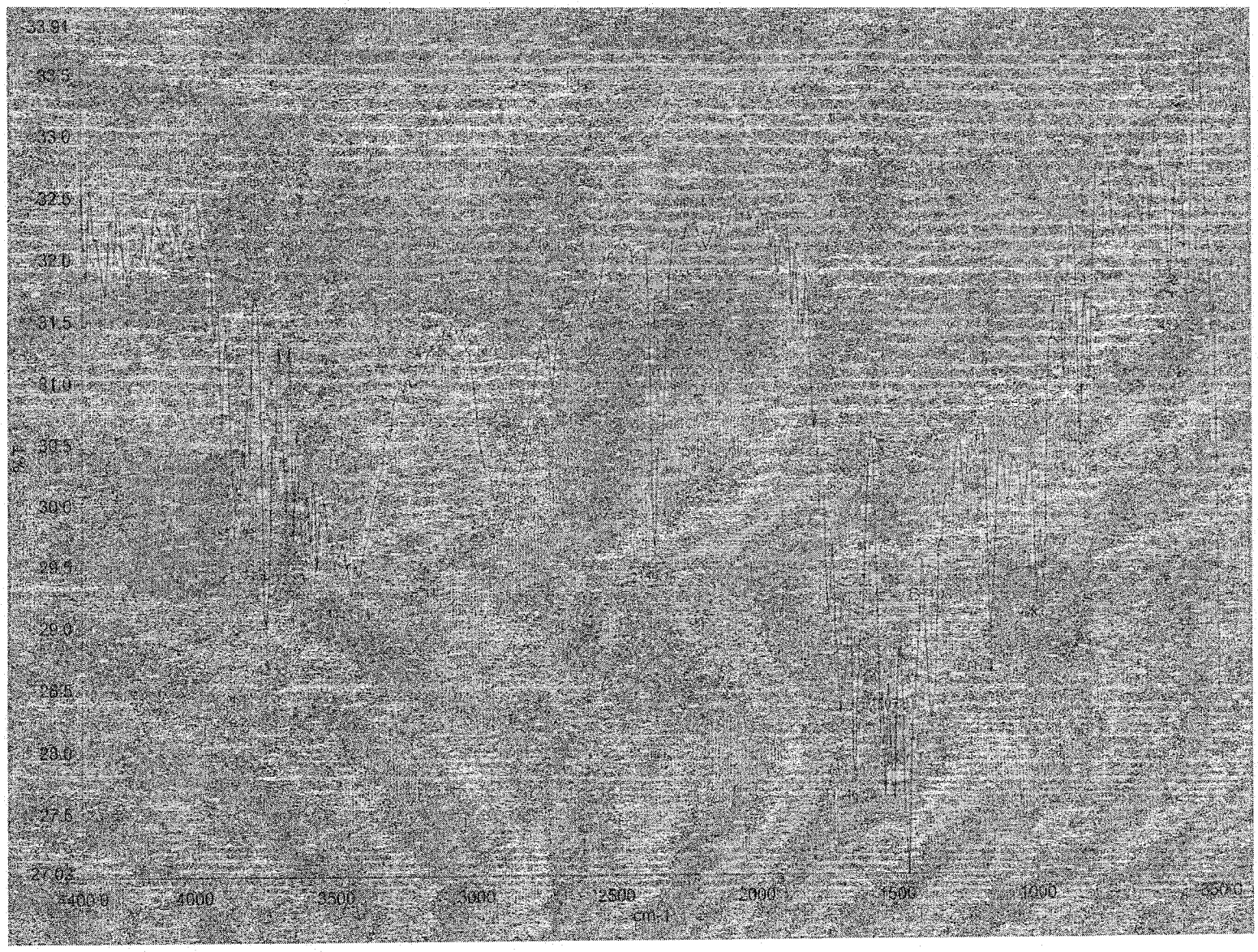


Figure (4.56) Transmission spectra of palm fiber (37%) /polypropylene (57%) with Epolene E-43 as compatibilizer.

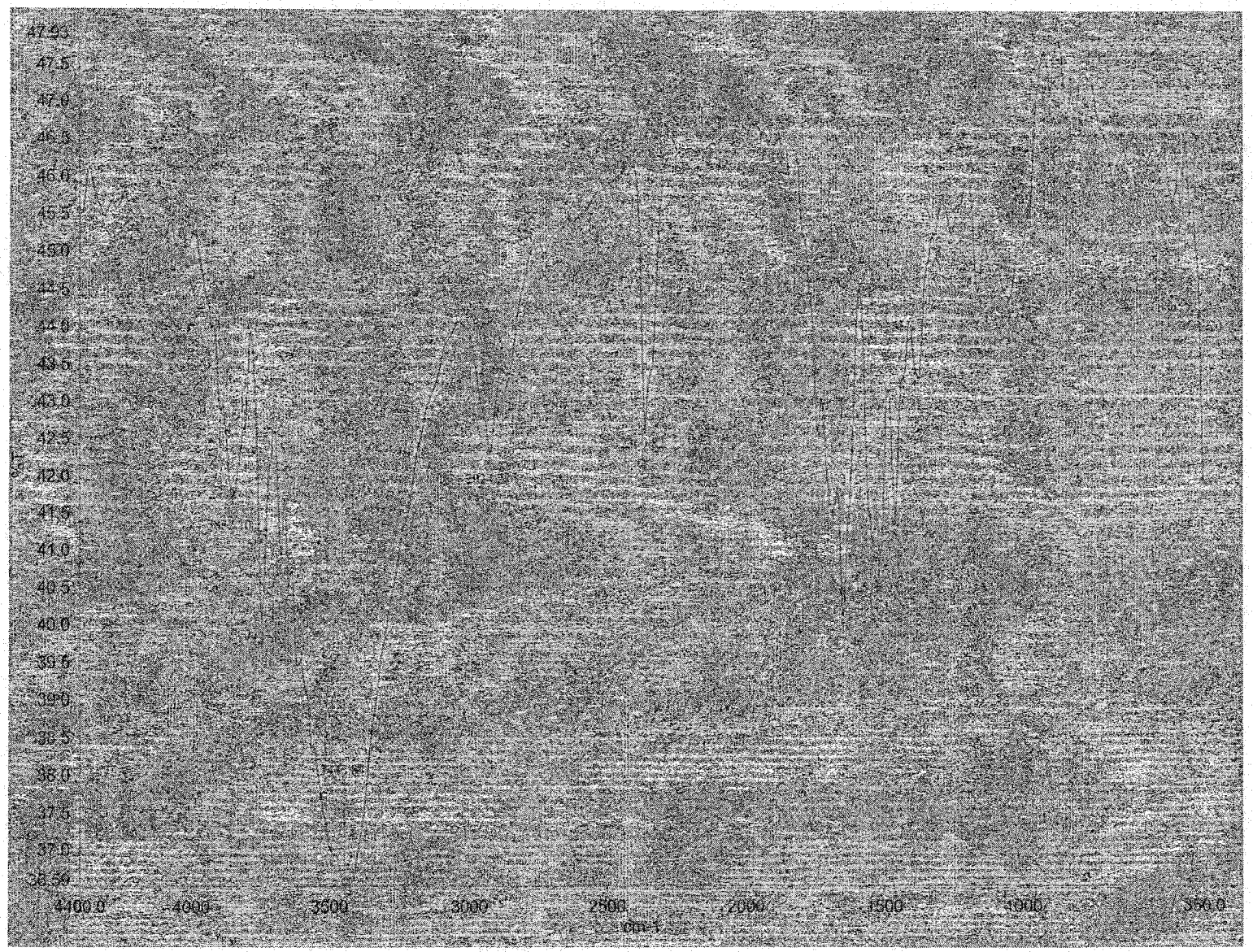


Figure (4.57) Transmission spectra of palm fiber (37%) / polypropylene (57%) with Epolene G-3003 as compatibilizer.

CHAPTER FIVE

CONCLUSION

It was found out that as the fiber content increases the composite modulus also increases which is an indication for the existence of adhesion to some degree between polypropylene and much stiffer palm fiber. However, the adhesion is not satisfactory resulting in decrease in composite tensile strength with fiber addition. In order to decrease the incompatibility between the polypropylene and the palm fiber, compatibilizer Epolene E-43 and Epolene G-3003 were incorporated into the composite. Mechanical testing showed that with the increase in compatibilizer content, the tensile strength improved. It was also found out that 6% of Epolene G-3003 resulted in the best composite performance.

Palm fiber / polypropylene composites containing no compatibilizer resulted in rough fracture surfaces with extensive fiber pullout which can be an indication for poor bonding between the palm fiber and polypropylene matrix.

Upon incorporation of the compatibilizer Epolene E-43 we saw that instead of fiber pullout, a smoother fracture surface (still with some roughness) is observed. Epolene E-43 seemed to have enhanced the interfacial adhesion between the fibers and matrix. That is why a higher load was transferred onto the fibers, hence a higher composite strength

was observed. Composites with compatibilizer Epolene G-3003 produced even smoother fracture surfaces with no significant fiber pullout. This is consistent with the results of the mechanical tests performed on the uncompatibilized and compatibilized composites. Composites with compatibilizer G-3003 were the strongest.

Differential Scanning Calorimetry was performed to see if the addition of compatibilizer enhanced the crystallinity of the composite. It was found out that addition of compatibilizer does not significantly improve the crystallinity of the composite.

Fourier Transform Infrared Spectroscopy was performed to study whether the addition of compatibilizer produced a new functional group in the composite, which would suggest chemical bonding between the fiber and the compatibilizer. As both compatibilizers were maelic anhydride grafted polypropylene. Formation of ester linkage was expected between the fiber and the compatibilizer that would show as an absorption around 1740 cm^{-1}. But the IR of the fiber exhibited a strong absorption at 1735cm^{-1} that could be attributed to some kind of carboxyl functional group. Hence it cannot be conclusively said that the addition of compatiblizer has formed new bonds with the palm fiber.

CHAPTER SIX

RECOMMENDATIONS

Following are the recommendations as a follow up to this work.

- An extruder that can apply higher rpm can be used to get even better dispersion of the fiber in the matrix.
- Different set of compatiblizers can be used. As a next step, maleic anhydride grafted polypropylene can also be prepared in the laboratory of the same brand of polypropylene which is used in this experiment, then added as a compatibilizer.
- Different brands of polypropylene with a lower melting temperature can be used to prepare these composite.
- This fiber can also be incorporated into other polymers like for example polyethylene. Separate study for polyethylene as the matrix can be conducted.

REFERENCES

Alagar, M., Dinakaran, K., Mahesh, K.P.O., and Kuman, A.A., "Studies On Thermal And Morphological Characteristics Of E-glass/Kevlar 49 Reinforced Siliconised Epoxy Composites", European Polymer Journal, 36, 2449-2454 (2000).

Angles, M.N., Salvado, J. and Dufresne, A., "Stream Exploded Residual Softwood-Filled Polypropylene Composites", Journal of Applied Polymer Science, 74, 1962-1977 (1999).

Belgacem, A.N., Bataille, P. and Sapieha, S., "Effect Of Corona Modification On The Mechanical Properties Of Polypropylene/Cellulose Composites", Journal of Applied Polymer Science, 53, 379-385 (1994).

Chodak, I. and Krupa, I., "Physical Properties Of Thermoplastic/Graphite Composites", European Polymer Journal, 37, 2159-2168(2001).

Coutinho, F.M.B., Costa, T.H.S., Suarez J.C.M. and Melo, D.P., "Sawdust Reinforced Polypropylene Composites : A Study Of Fracture Behaviour", Polymer Testing, 19, 625-633 (2000).

Feldman, D., Denes, F., Zeng Z., Denes, A.R., and Banu, D., " Polypropylene Fiber Matrix Bonds In Cementitious Composites", Journal of Adhesion Science & Technology, 14:13, 1705-1721 (2000).

Forest Products Laboratory, "Wood Handbook : Wood As An Engineering Material", University Press of the Pacific , 2000.

Fuad, M.Y.A., Yaakob, I., Rusli, O., Ishak, Z.A.M. and Omar, A. K. M. "Determination Of Filler Content In Thermoplastic Composites By FTIR Analysis", Journal of Applied Polymer Science, 56, 1557-1650 (1995).

Gan, D., Lu, S., Song, C. and Wang, Z., "Mechanical Properties And Frictional Behavior Of A Mica Filled Poly(aryl ether ketone) Composite", European Polymer Journal, 37, 1359-1365 (2001).

Gassan, J. and Blendzki, A.K., "Possibilities To Improve The Properties Of Natural Fiber Reinforced Plastics By Fiber Modification – Jute Polypropylene Composites –", Applied Composite Material, 7, 373-385 (2000).

Gilbert, R.D., "Cellulosic Polymers, Blends And Composites", Hansen Gardner Publications, 1994.

Harikumar, K.R., Joseph, K. and Thomas, S., "Jute Sack Cloth Reinforced Polypropylene Composites : Mechanical & Sorption Studies", Journal of Reinforced Plastics and Composites, 18:4, 346-372 (1999).

Manrich, S., "Studies On Recycling Of Post Consumer Plastics Waste For Substitutive Applications In Writable And Printable Paper", Polymer Recycling, 5, 213-221 (2000).

Mwaikambo, L. Y., Martuscelli, E. and Avella, M., "Kapok/Cotton Fabric-Polypropylene Composites", Polymer Testing, 19, 905-918 (2000).

Nagura, M., Ohkoshi, Y. and Gotoh, Y., "Preparation And Optical Absorption Property Of Poly(acrylic acid)/Copper Sulfide Nanocomposite Films", Polymer Journal, 33, 303-305 (2001).

Okazaki, M., Maruyama, K., Tsuchida, M., and Tsubokawa, N., " A Novel Gas Sensor From Poly(ethylene glycol)- Grafted Carbon Black. Responsibility Of Electric Resistance Of Poly(ethylene glycol)-Grafted Carbon Black Against Humidity And Solvent Vapor", Polymer Journal, 31 : 8, 672-676 (1999).

Park, B.D. and Balatinecz, J., "Short Term Flexural Creep Behavior Of Wood-Fiber/Polypropylene Composites", Polymer Composites, 19:4, 377-382 (1998).

Patil, R.C., Ahmed, S.M. and Ogura, K., "Preparation And Characterization Of Highly Transparent Conduction Composites Consisting Of Acid Doped Poly(o-toluidine) And Poly(methylmethacrylate)", Polymer Journal, 32 : 6, 466-470 (2000).

Quillin, D., Yin, M., Koutsky, J.A. and Caulfield, D.F., "Crystallinity in the Polypropylene/Cellulose System. II. Crystallization Kinetics", Journal of Applied Polymer Science, 52, 605-615 (1994).

Raj, R.G., Kokta, B.V., "Compounding Of Cellulose Fibers With Polypropylene : Effect Of Fiber Treatment On Dispersion In The Polymer Matrix", Journal of Applied Polymer Science, 38, 1987-1996 (1989).

Rana, A.K., Mandal, A., Mitra, B.C., Jacobson, R., Rowell, R. and Banerjee, N., "Short Jute Fiber-Reinforced Polypropylene Composites: Effect Of Compatibilizer", Journal of Applied Polymer Science, 69, 329-338 (1998).

Rowell, R.M., Young, R.A. and Rowell, J.K., "Paper And Composites From Agro Based Resources". Lewis Publishers, 1996.

Rozman, H. D., Tan, K. W., Kumar, R. N. and Abubakar, A., "Preliminary Studies on the Use of Modified ALCELL Lignin as a Coupling Agent in the Biofiber Composites", Journal of Applied Polymer Science, 81, 1333-1340 (2001b).

Rozman, H.D., Tan, K.W., Kumar, R.N., Abubakar, A., Ismail, H.and Ishak, Z.A.M., "The Effect Of Lignin As A Compatibilizer On The Physical Properties Of Coconut Fiber-Polypropylene Composites", European Polymer Journal, 36, 1483-1494, (2000).

Rozman, H.D., Tay, G.S., Kumar, R.N., Abusamah, A., Ismail, H. and Ishak, Z.A.M., "Polypropylene-Oil Palm Empty Fruit Bunch-Glass Fiber Hybrid Composites : A Preliminary Study On The Flexural And Tensile Properties", European Polymer Journal, 37, 1283-1291 (2001a).

Rozman, H.D., Tay, G.S., Kumar, R.N., Abusamah, A., Ismail, H. and Ishak, Z.A.M., "Polypropylene Hybrid Composites : A Preliminary Study On The Use Of Gass And Coconut Fiber As Reinforcements In Polypropylene Composites", Polymer Plasitc Technology Engineering, 38, 997-1011 (1999).

Sain, M. M. and Kotka, B.V., "Study On Molecular Interaction In Polyfunctional Maleimide Modified Polypropylene/Pulp Composites by ESCA, FTIR, & DSC", Journal of Adhesion Science & Technology, 7:7, 743-760 (1993).

Schwartz, M.M., "Composite Material Handbook", 2nd Edition, MacGraw Hill Inc, New York, 1992.

Son, S. J., Lee, Y.M. and Im, S.S., "Transcrystalline Morphology And Mechanical Properties In Polypropylene Composites Containing Cellulose Treated With Sodium Hydroxide And Cellulose", Journal of Material Science, 35, 5767-5778 (2000).

Stark, N.M., "Wood Fiber Derived From Scrap Pallets Used In Polypropylene Composites", Forest Products Journal, 49:10, 39-46 (1999).

Weiss, R.A. and Jesus, M.C., "Synthesis Of Conductive Nanocomposite By Selective In situ Polymerization Of Pyrrole Within Lamellar Microdomains Of A Block Copolymer", Macromolecules, 31, 2230-2235 (1998).

Wu, C.C. and Huang, C.Y., "The EMI Shielding Effectiveness Of PC/ABS/nickel Coated Carbon Fiber Composites", European Polymer Journal, 36, 2729-2737 (2000).

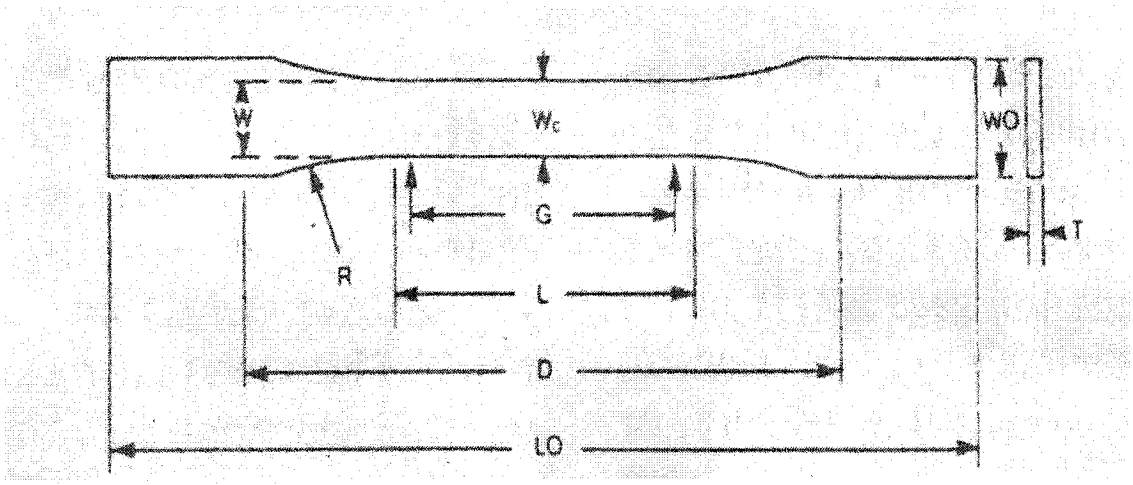
Xiaoyu, L., Benson, R. S., Kit, K. M. and Dever, M. "Kudzu Fiber Reinforced Polypropylene Composite", Journal of Applied Polymer Science, 85, 1961-1969(2002).
Yam, K.L., Gogoi, B.K., Lai, C.C. and Selke, S.E., "Composites From Compounding Wood Fibers With Recycled High Density Polyethylene", Polymer Engineering and Science, 30:11, 693-699 (1990).

Younguist, J.A., " Unlikely Partners ? The Marriage Of Wood And Non Wood Materials", Forest Products Journal, 45:10, 25-30 (1995).

Zafeiropoulos, N.E., Baillie, C.A. and Matthews, F.L. "A Study Of Transcrystallinity And Its Effect On The Surface On The Interface In Flax Fibre Reinforced Composite Materials", Composites Part A, 32, 525-543 (2001).

APPENDICES

Section A



Where

W	Width of narrow section	= 6 mm
L	Length of narrow section	= 57 mm
WO	Width overall	= 19 mm
LO	Length overall	= 183 mm
G	Gage length	= 50 mm
D	Distance between grips	= 135 mm
R	Radius of fillet	= 76 mm

Figure (A1) Sketch of Tensile Specimen.

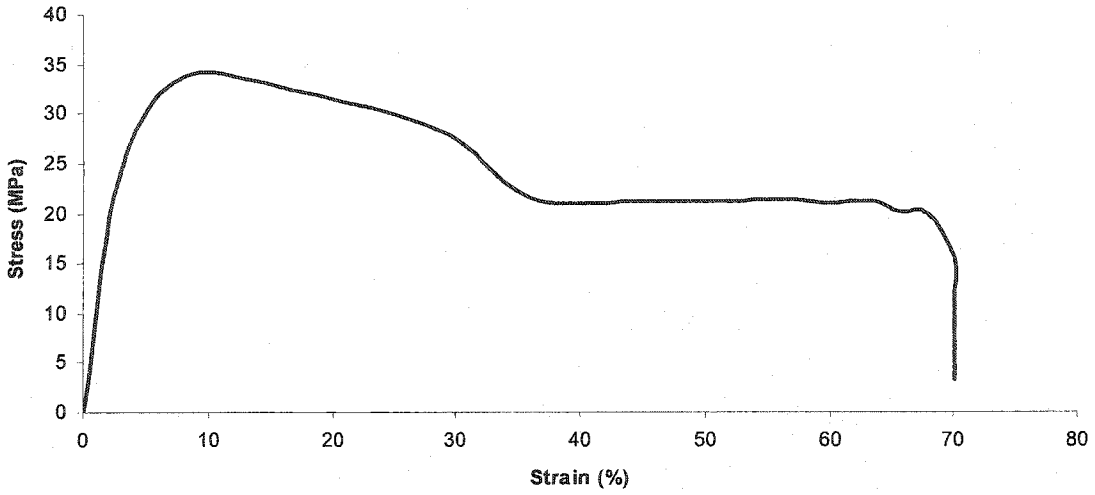


Figure (A2) Pure Polypropylene (Stress/Strain curve)

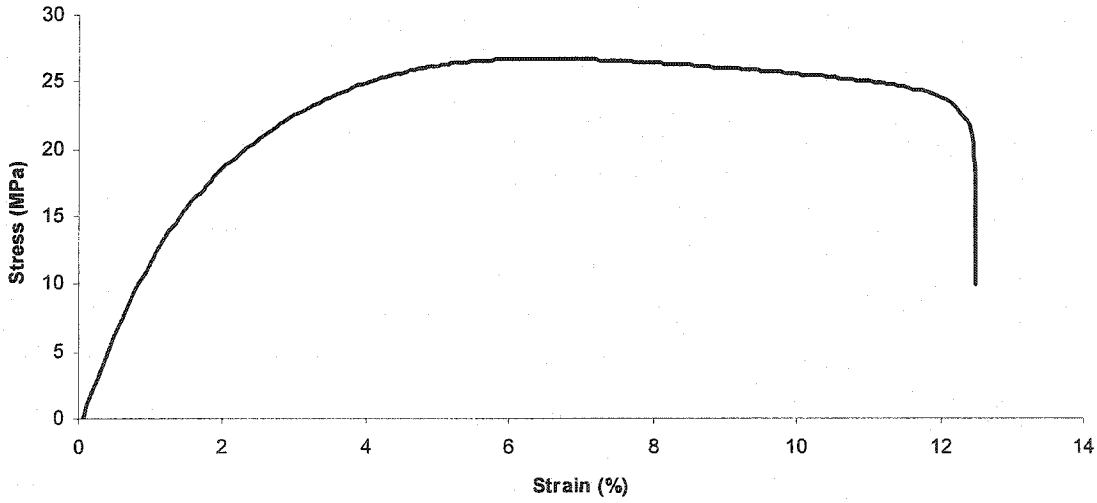


Figure (A3) PP/20 wt% fiber (Stress/Strain Curve).

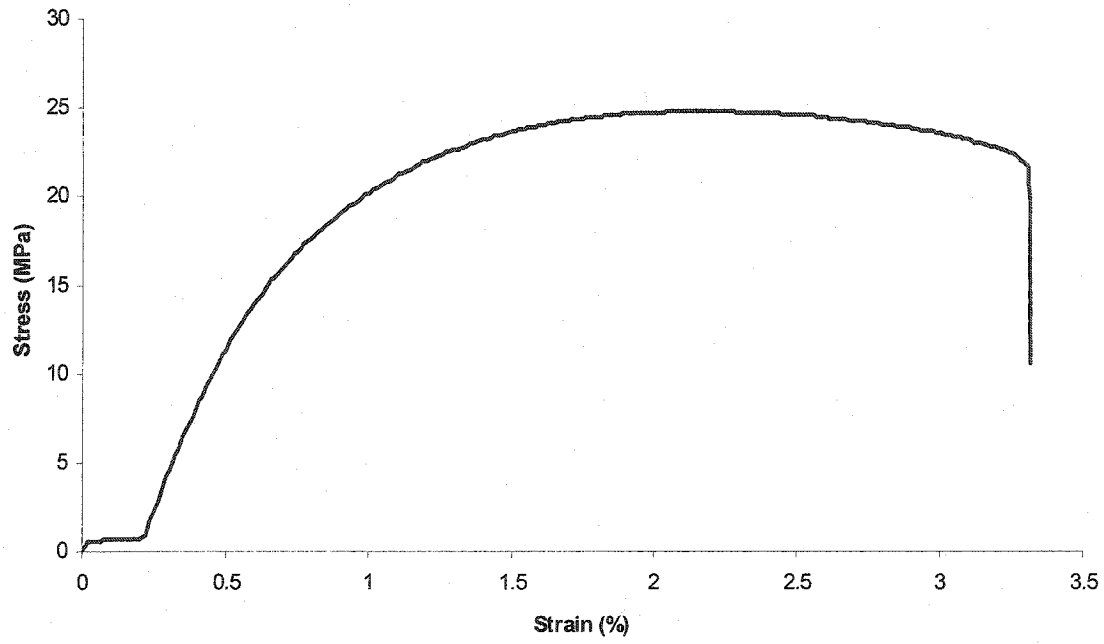


Figure (A4) PP/30 wt% fiber (Stress/Strain Curve)

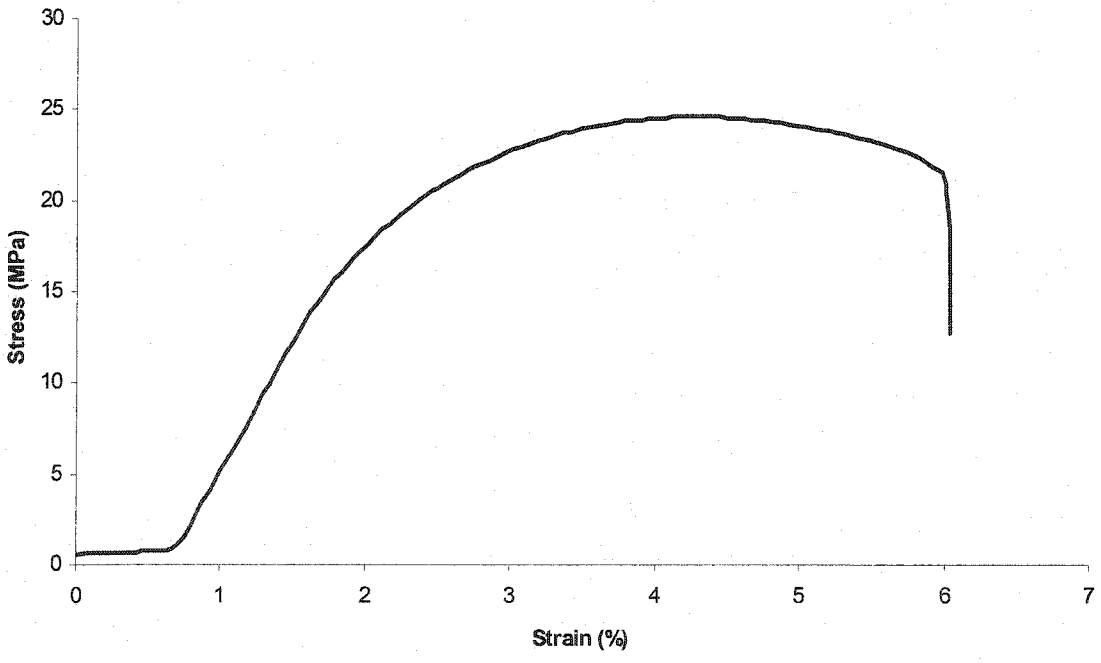


Figure (A5) PP/40 wt% fiber (Stress/Strain Curve).

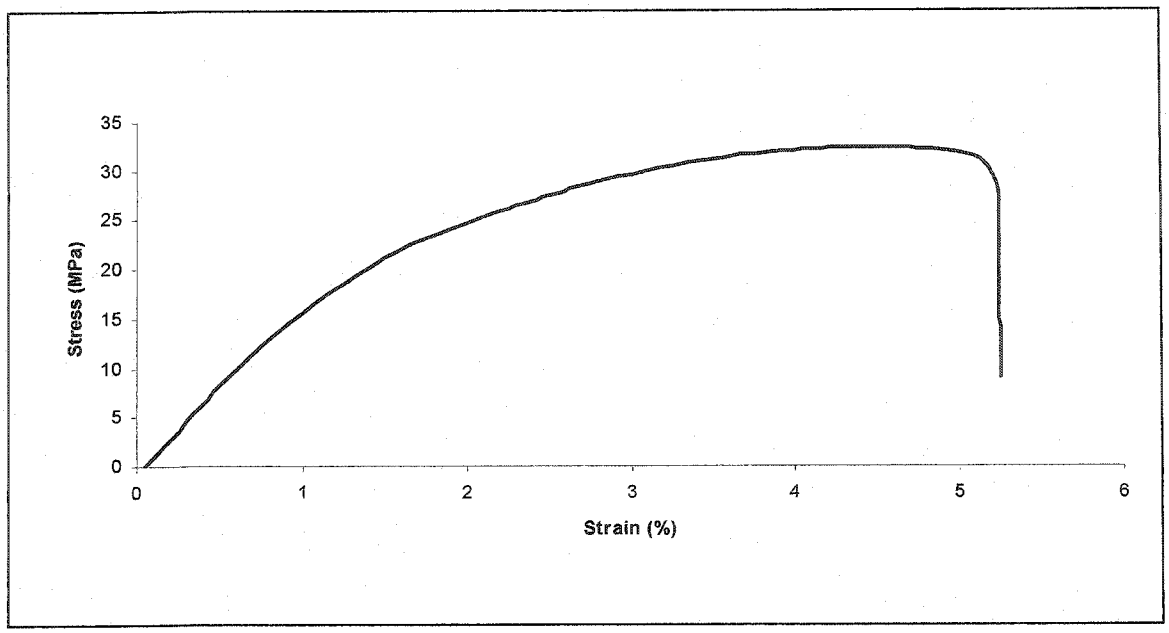


Figure (A6) 6 wt% G-3003 in PP/40 wt% fiber (SS Curve).

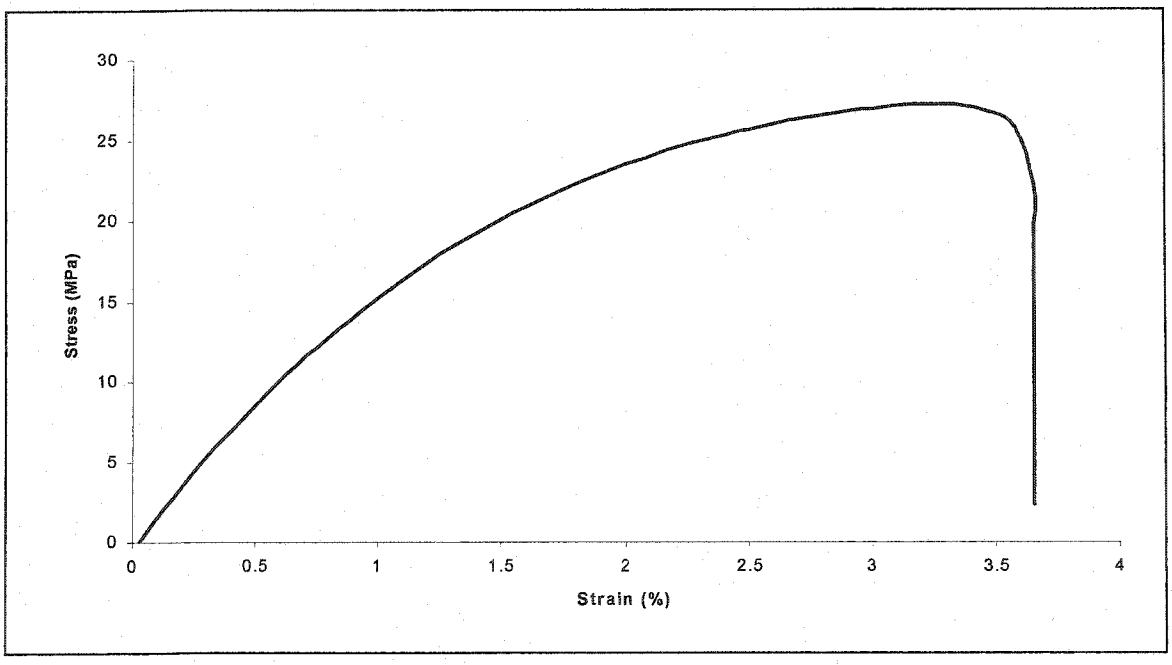


Figure (A7) 6% EP-43 added in PP/40 wt% fiber (SS Curve).

Section B

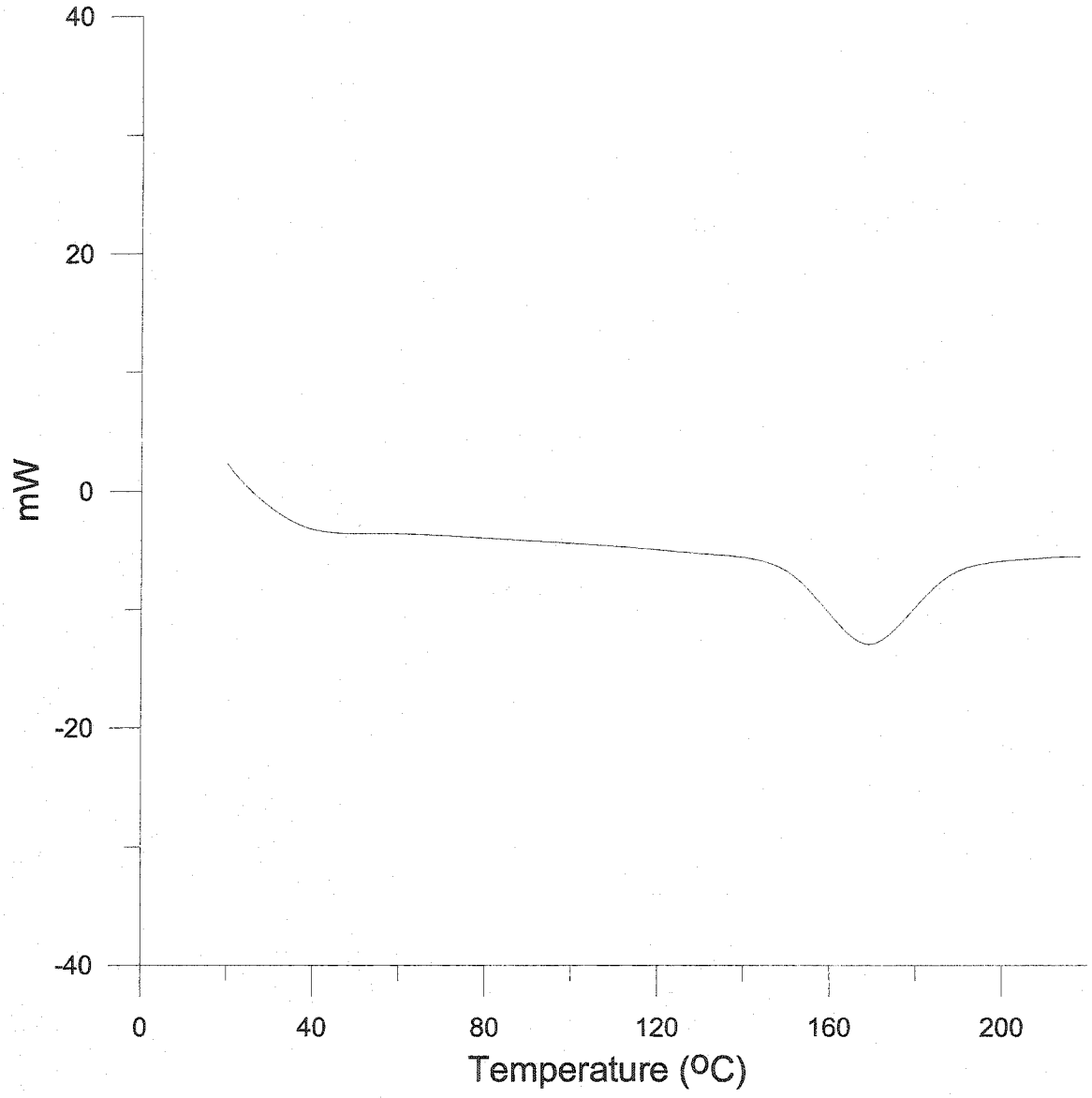


Figure (B1) Thermogram of Pure Polypropylene.

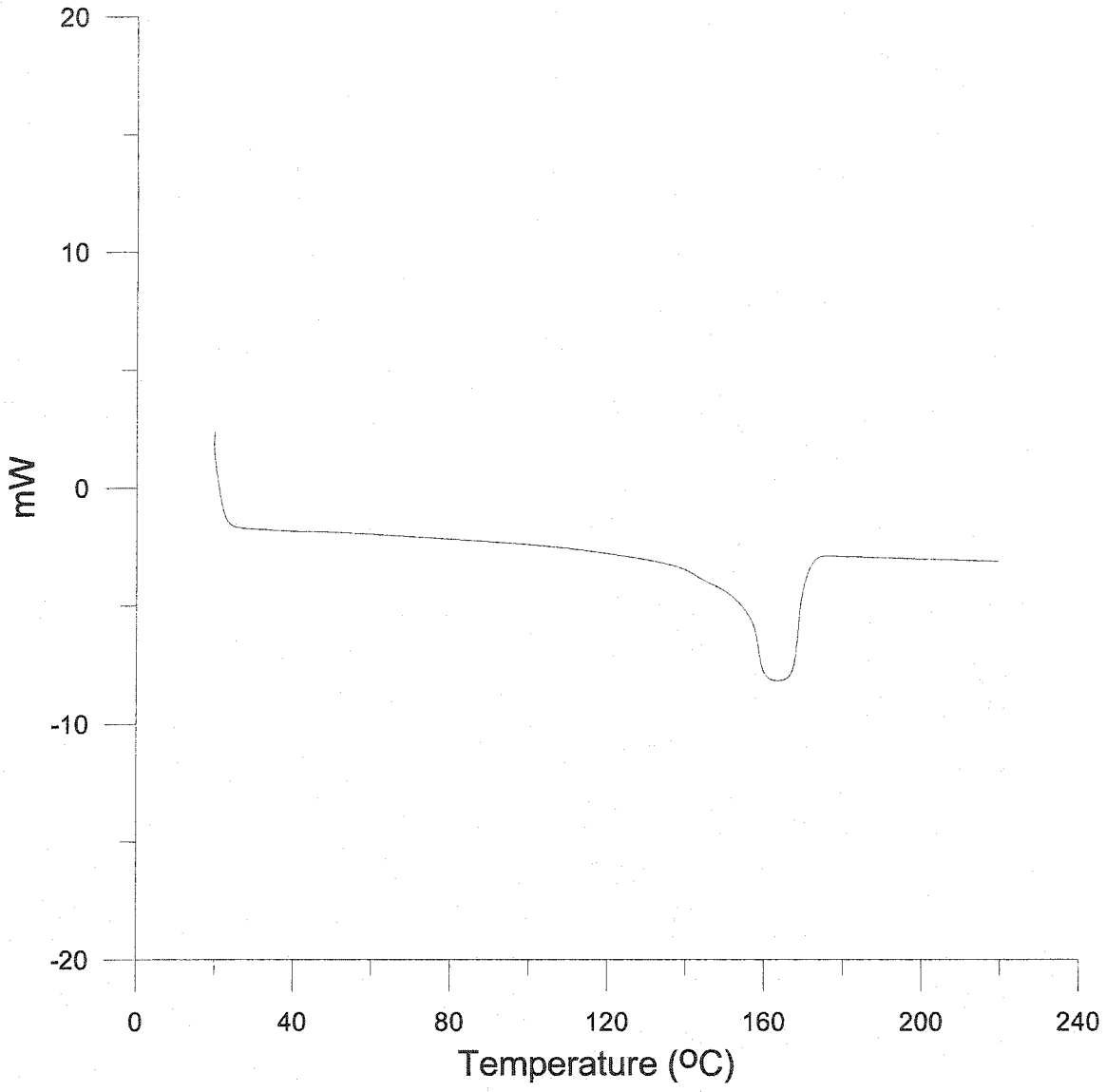


Figure (B2) Thermogram of palm fiber (20%) / polypropylene (80%) composite.

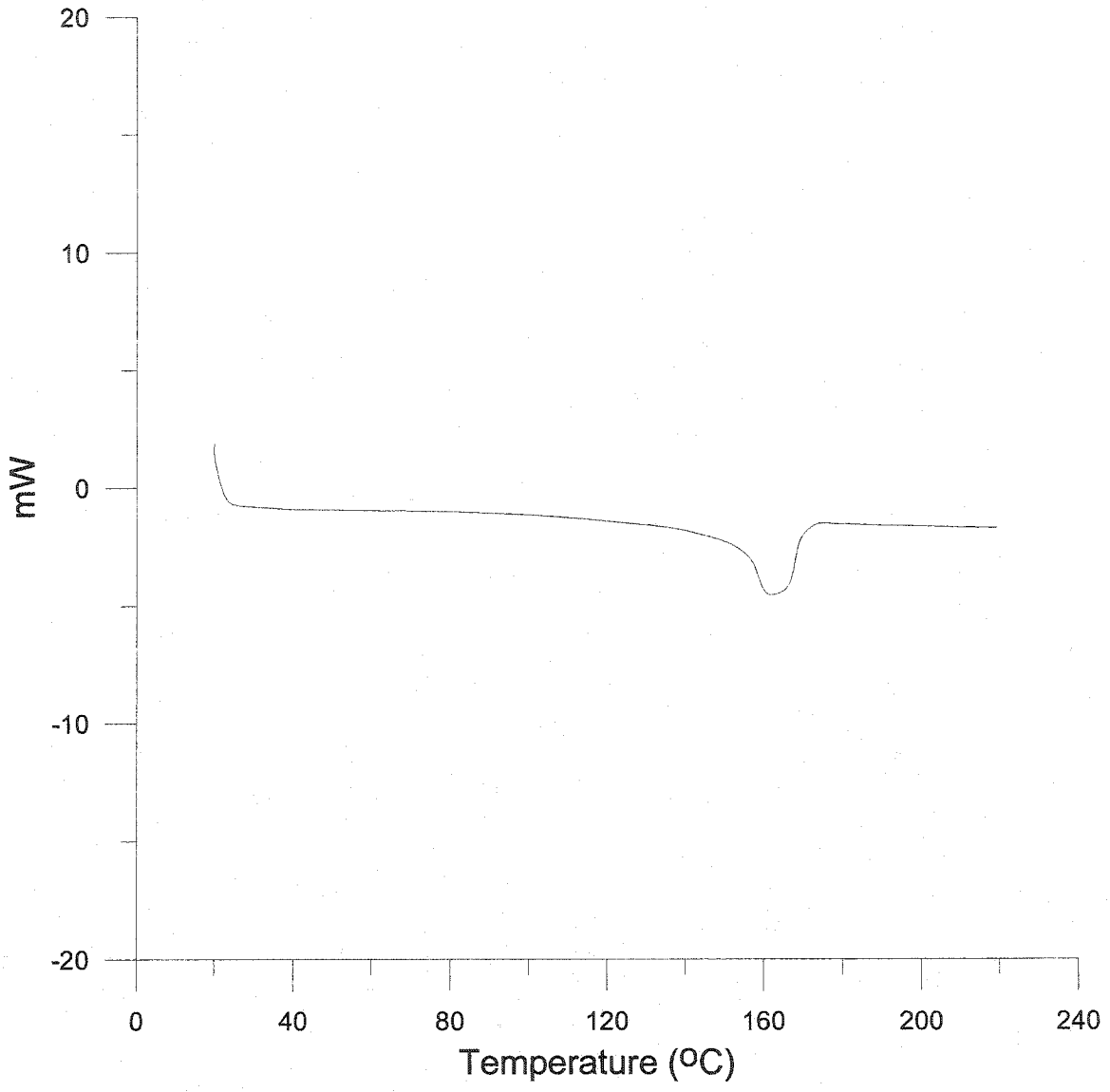


Figure (B3) Thermogram of palm fiber (30%) / polypropylene (70%) composite.

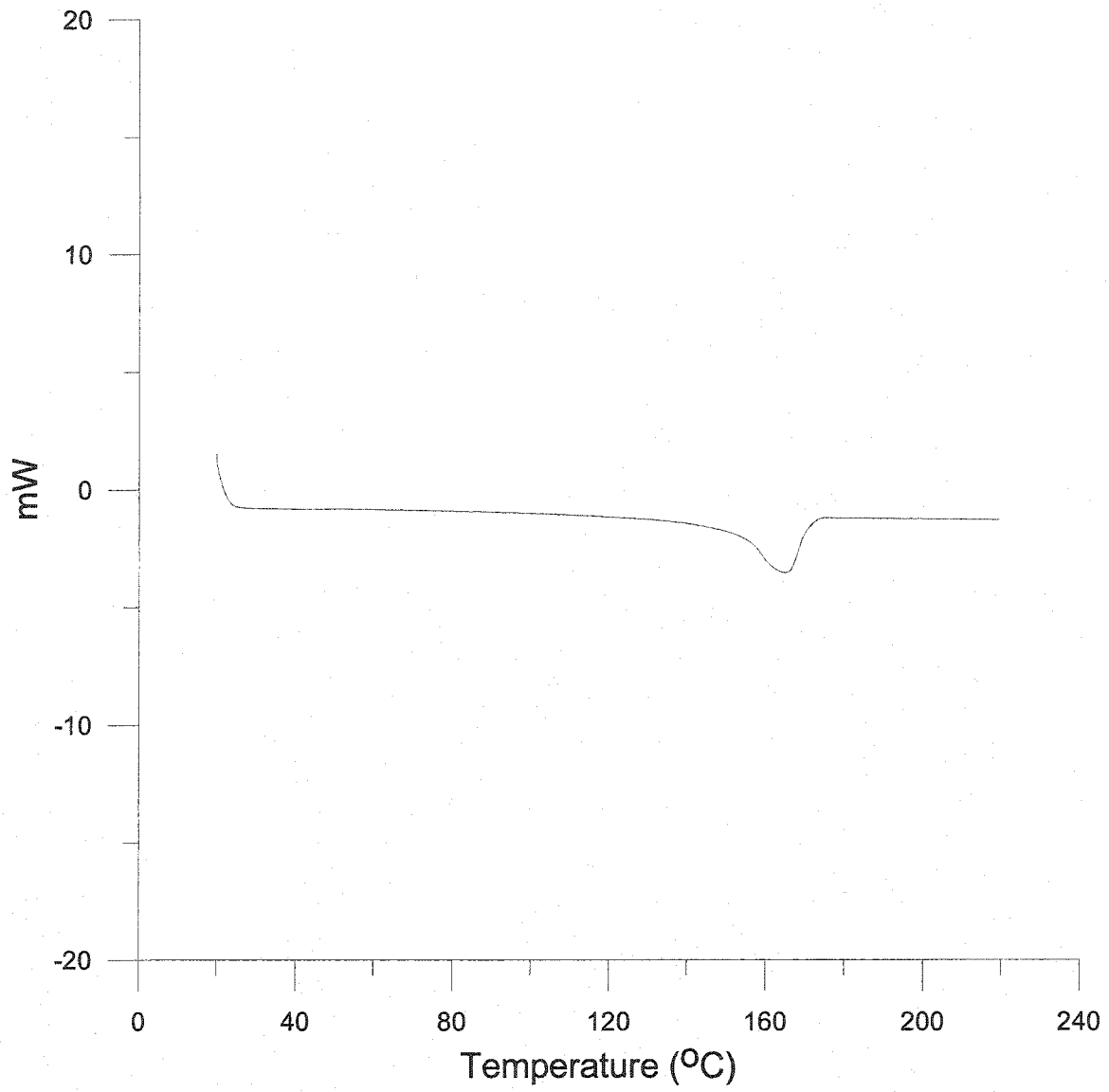


Figure (B4) Thermogram of palm fiber (40%) / polypropylene (60%) composite.

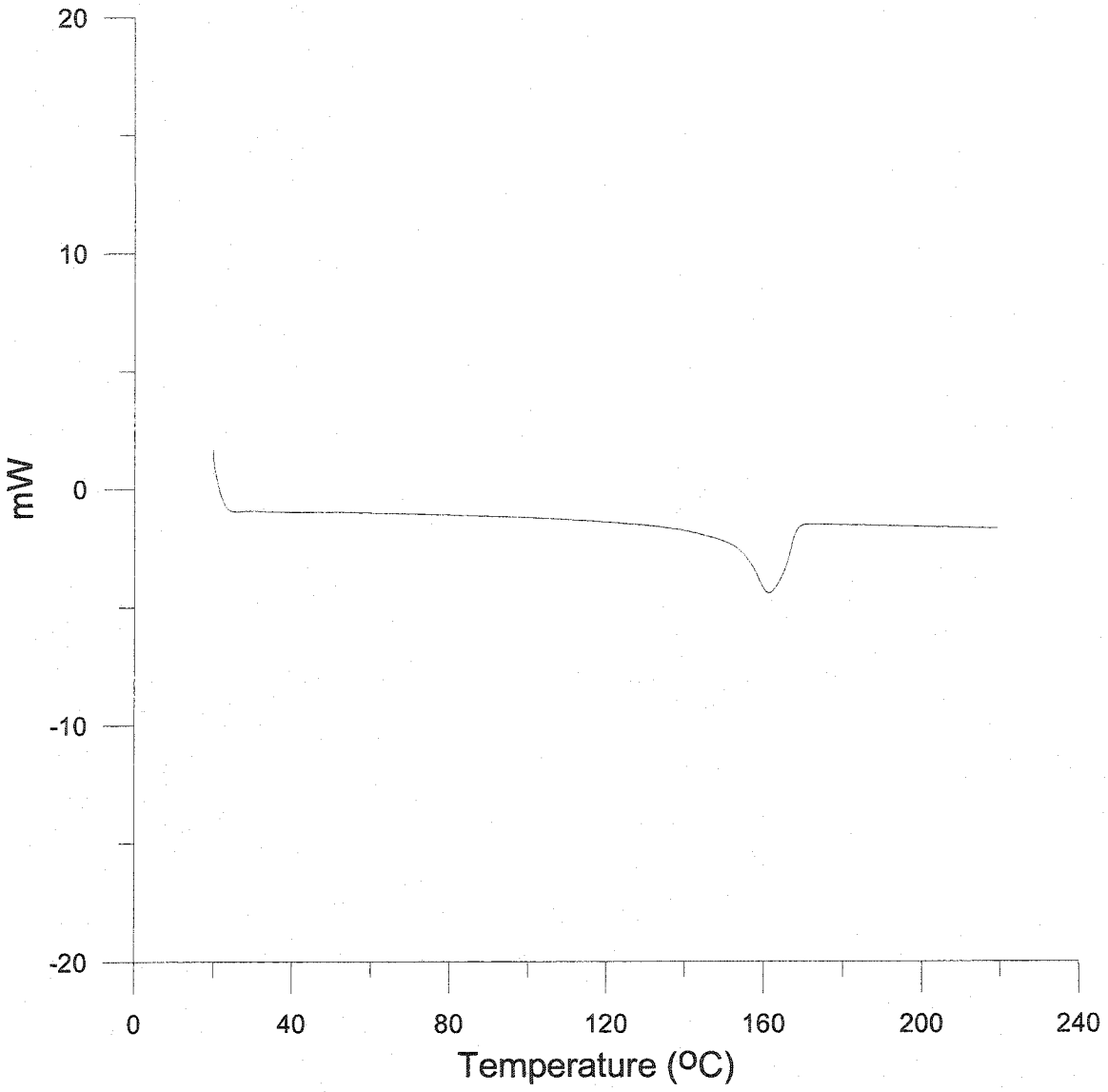


Figure (B5) Thermogram of palm fiber (40%) / polypropylene (60%) composite with 2% Epolene E-43.

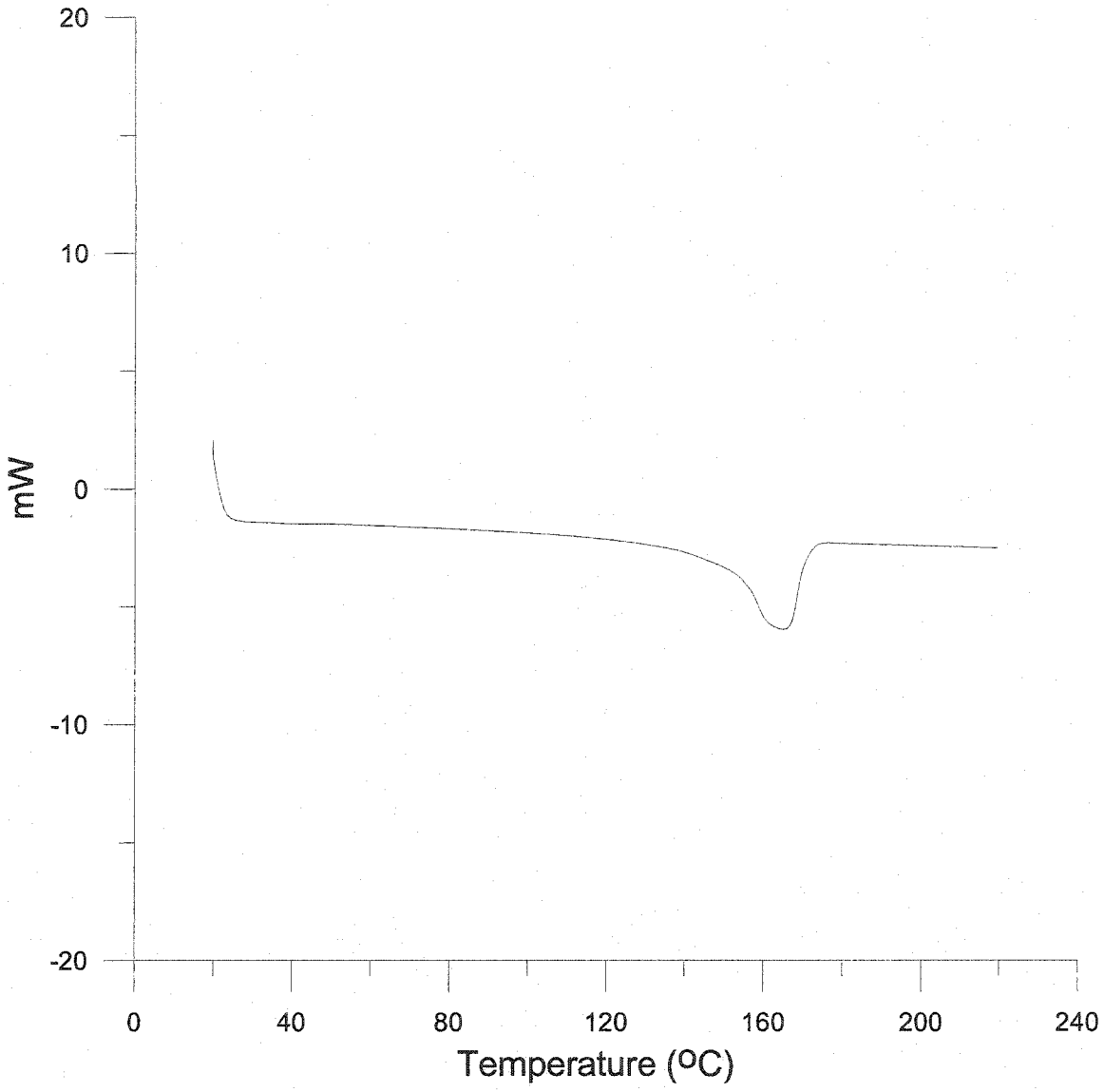


Figure (B6) Thermogram of palm fiber (40%) / polypropylene (60%) composite with 4% Epolene E-43.

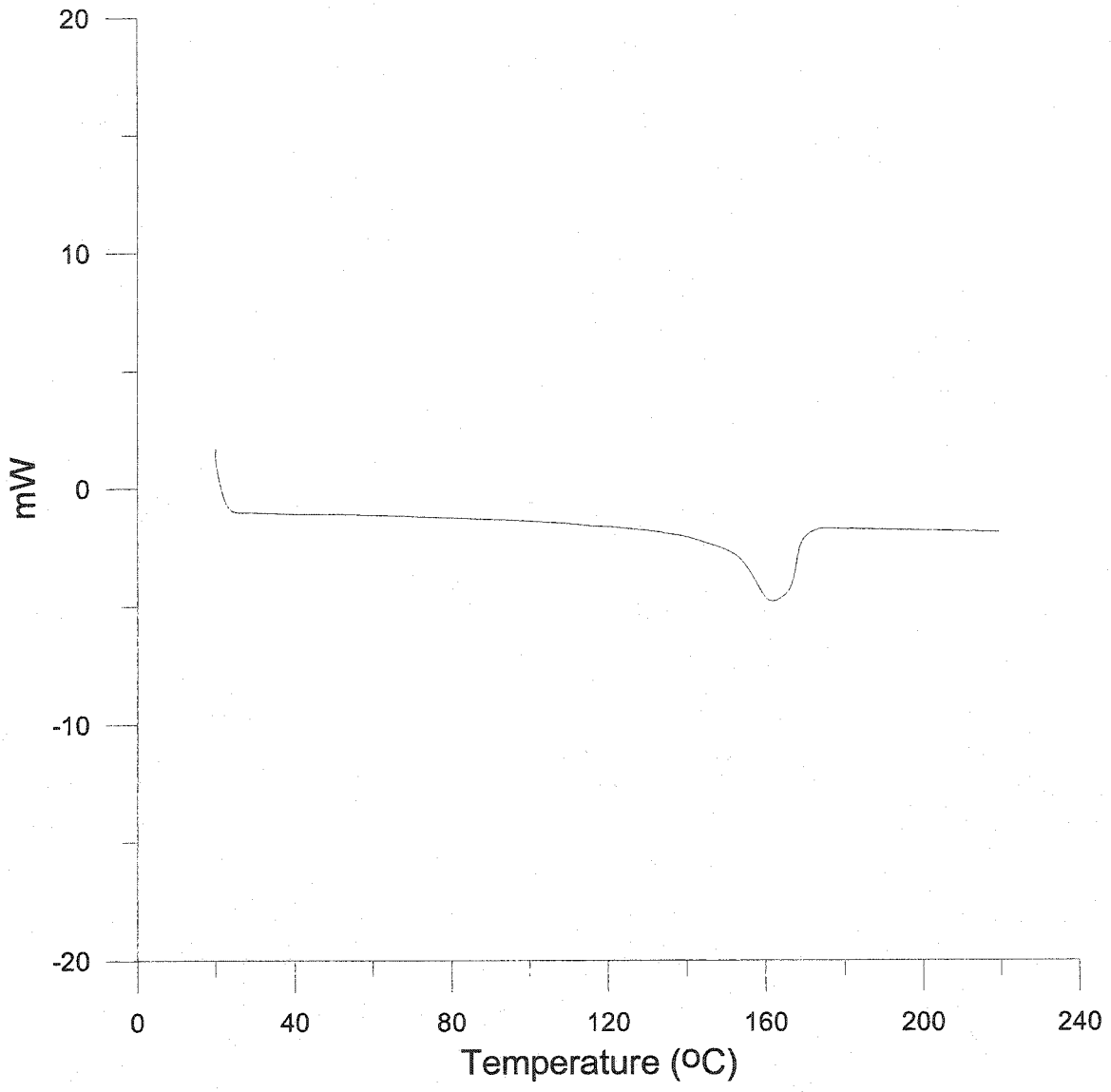


Figure (B7) Thermogram of palm fiber (40%) / polypropylene (60%) composite with 6% Epolene E-43.

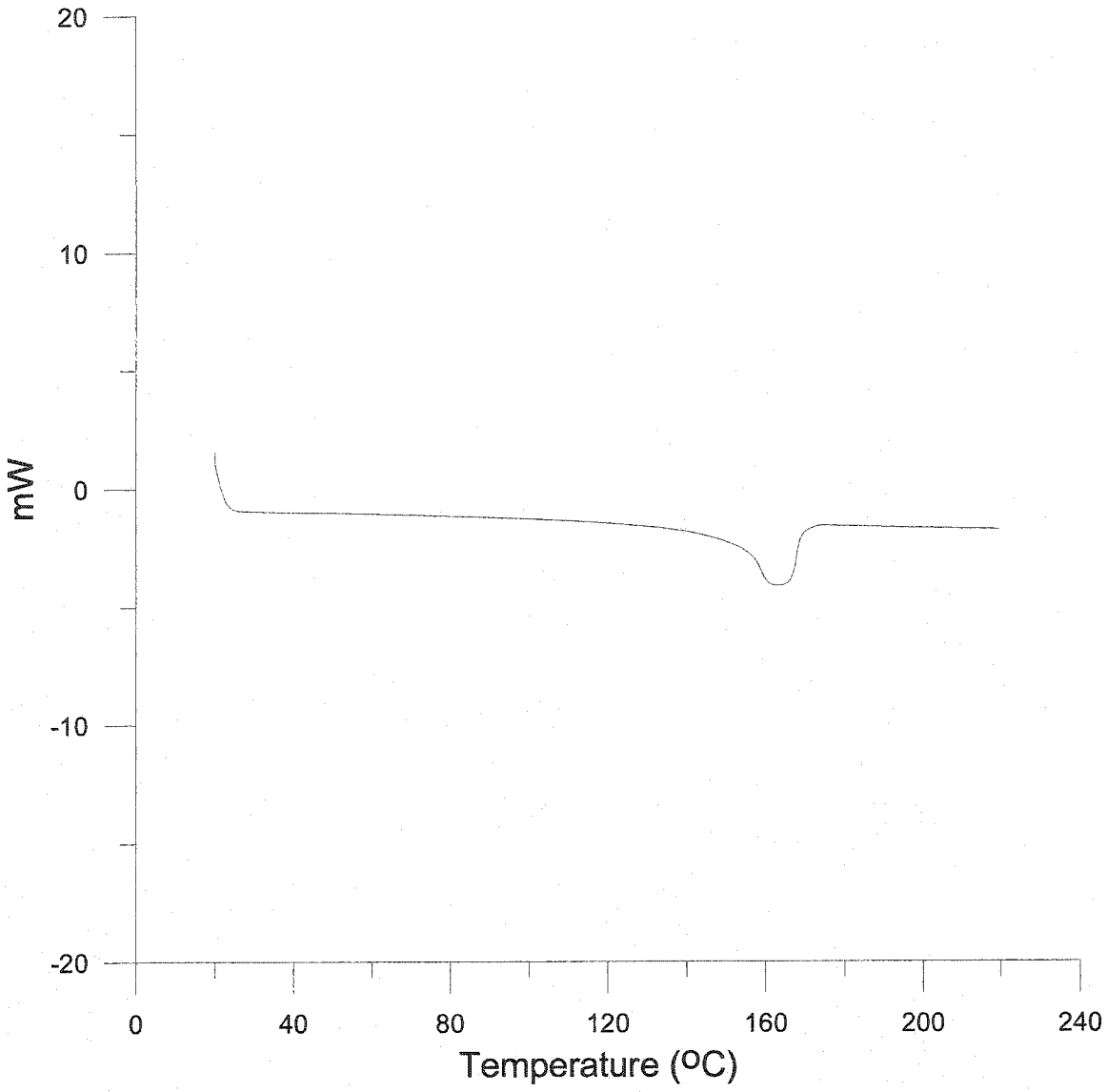


Figure (B8) Thermogram of palm fiber (40%) / polypropylene (60%) composite with 2% Epolene G-3003.

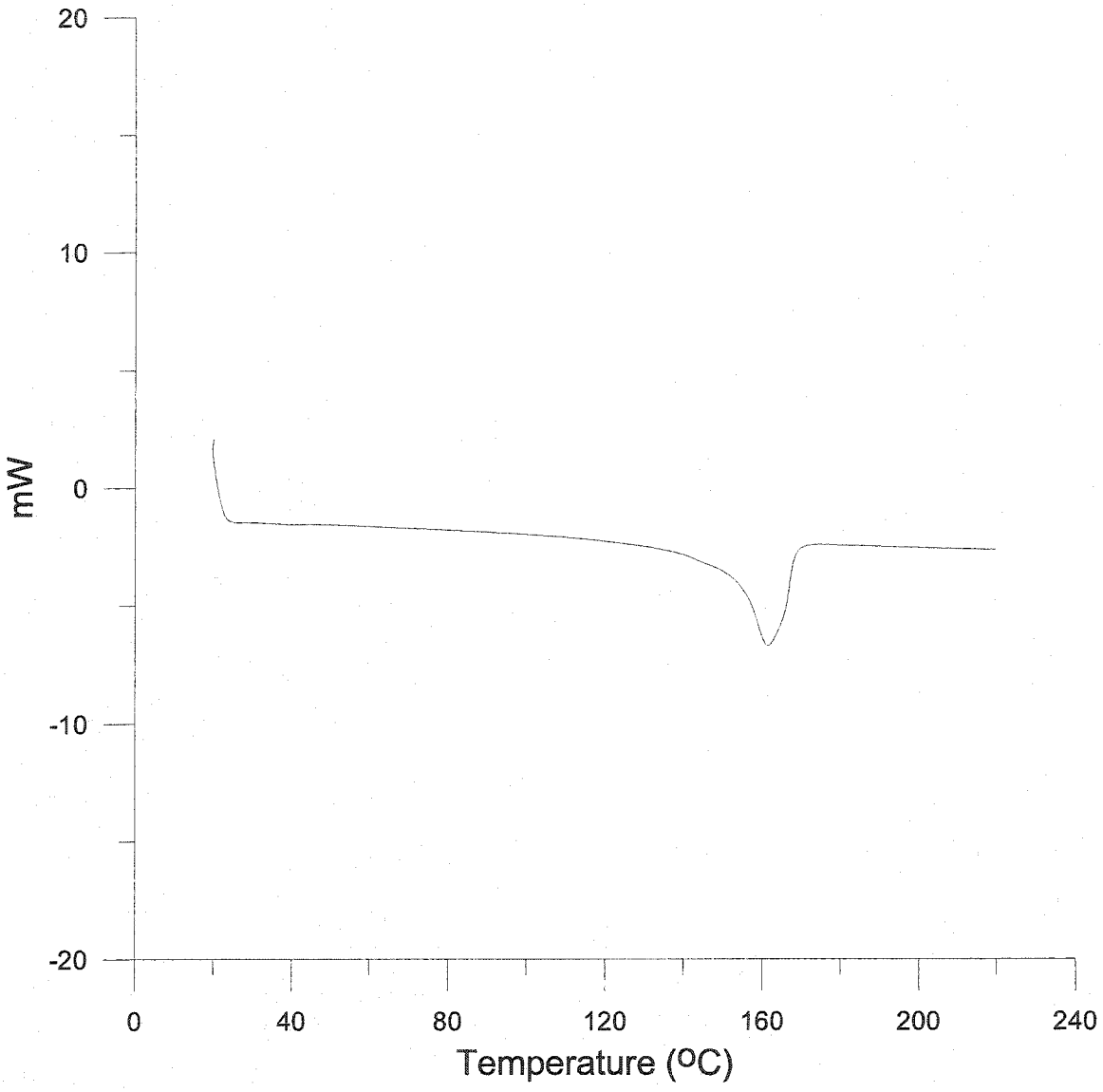


Figure (B9) Thermogram of palm fiber (40%) / polypropylene (60%) composite with 4% Epolene G-3003.

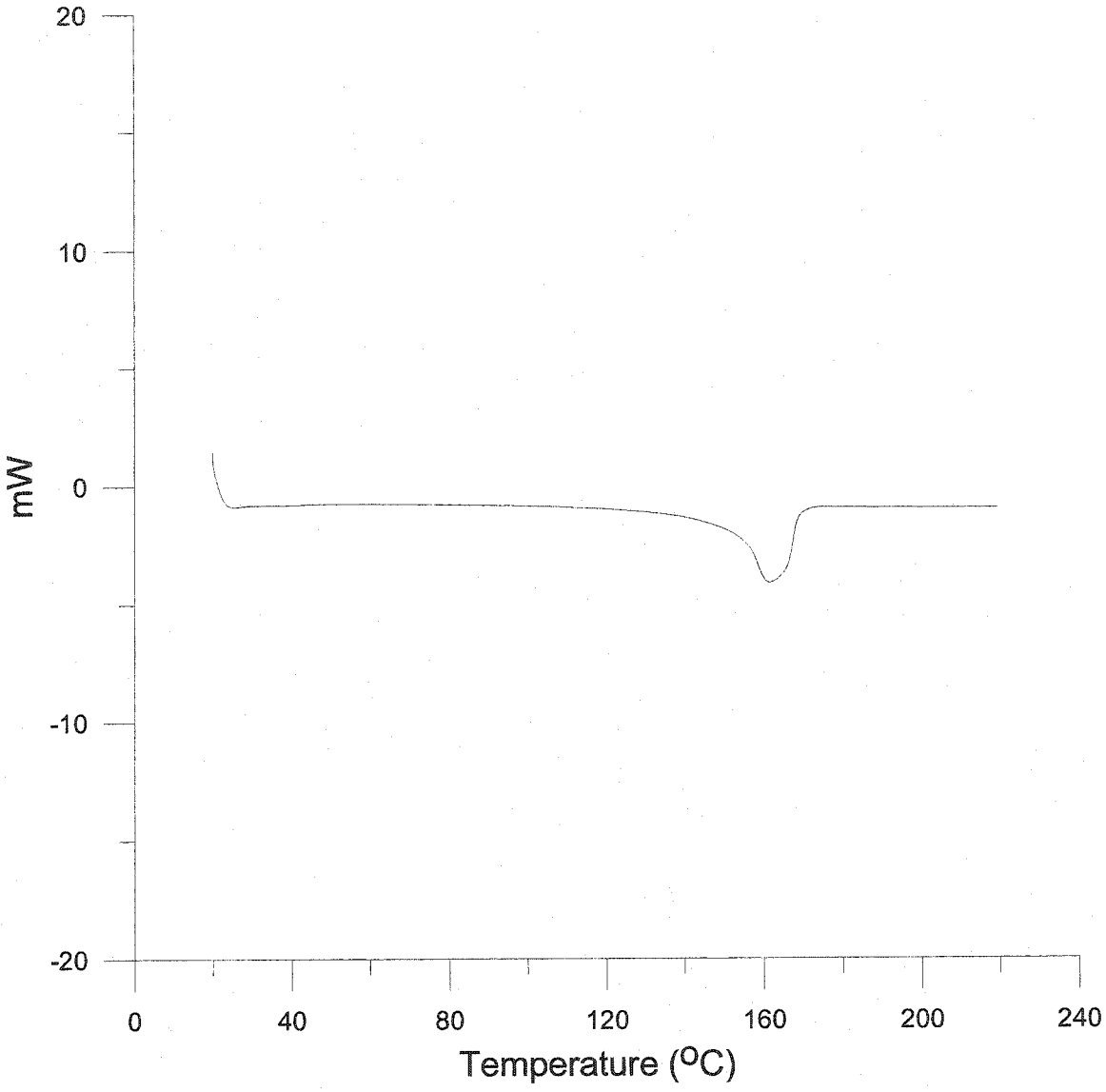


Figure (B10) Thermogram of palm fiber (40%) / polypropylene (60%) composite with 6% Epolene G-3003.

VITA

Name	SARFRAZ HAIDER ABBASI
Date & Place of Birth	11 August 1975, Dadu (PAKISTAN)
Nationality	PAKISTANI
Religion	ISLAM
Educational Qualification	B.Sc in Applied Chemistry from University of Karachi (1995-1997).
	M.Sc. in Applied Chemistry from University of Karachi (1998-1999).
	M.S. in Chemical Engineering from King Fahd University of Petroleum and Minerals (2001-2003).